Los Alamos Research Park Coated Conductor Development

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Superconductivity Technology Center
Los Alamos National Laboratory

FY2003 Funding: $1.4M; 4 FTE

LANL Coated Conductor Development

10 years

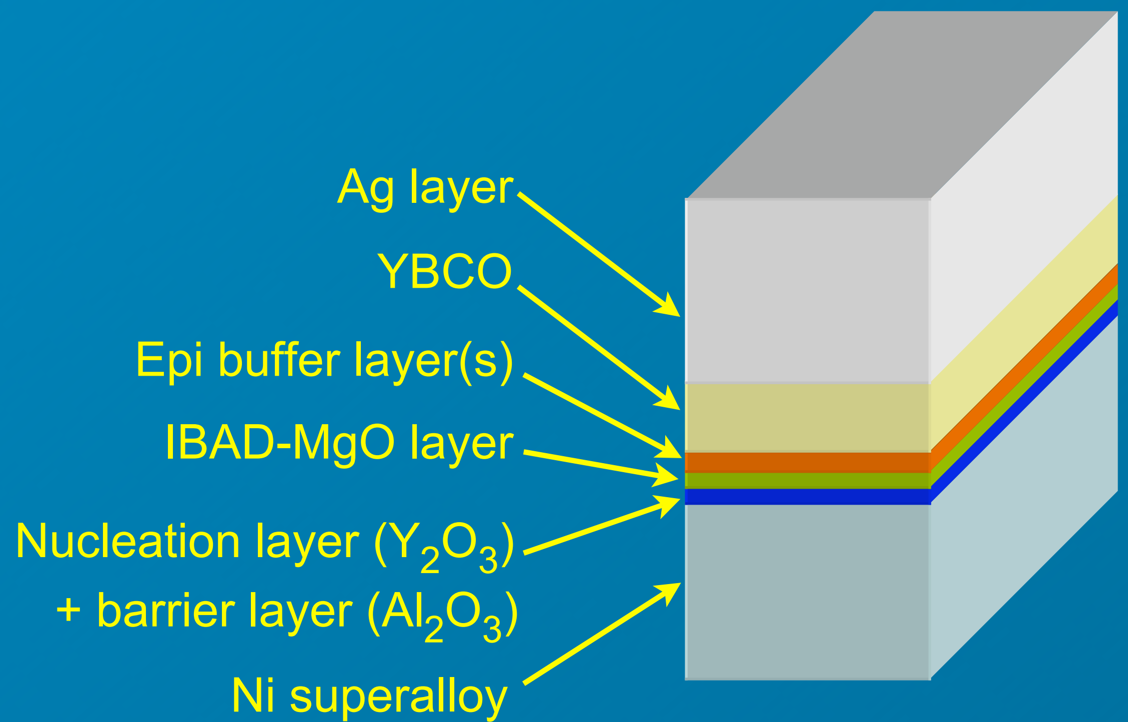
2 years
Year 1: 2.5 FTE
Year 2 : 4 FTE
$2M capex

Research Park

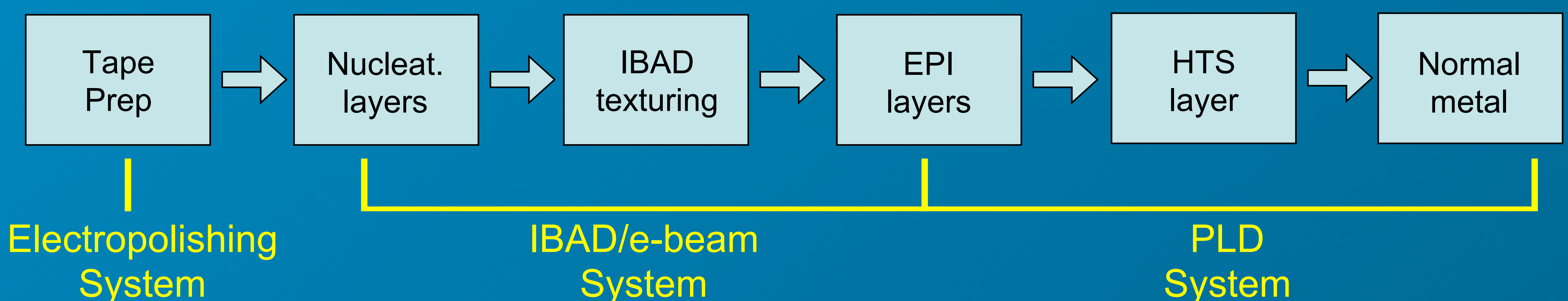
- Scale up fabrication & characterization (reel-to-reel)
- Increase interaction with outside collaborators
- Provide longer samples to collaborators
- Provide process specifications to industry:
  - Possible architectures
  - Inputs for cost analysis
- New *in situ* diagnostic capabilities
- Provide a unique facility that is available to users

LANL Coated Conductor

- IBAD-textured MgO template on a Ni-superalloy
  - IBAD layers deposited by e-beam evaporation at RP
- Pulsed-laser deposited buffers and superconductor

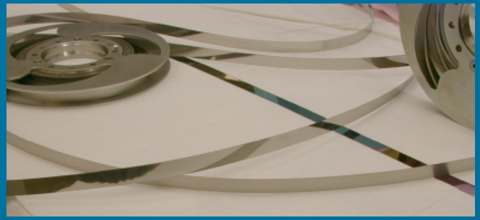


Los Alamos Research Park:



Los Alamos Research Park Labs: Year 2

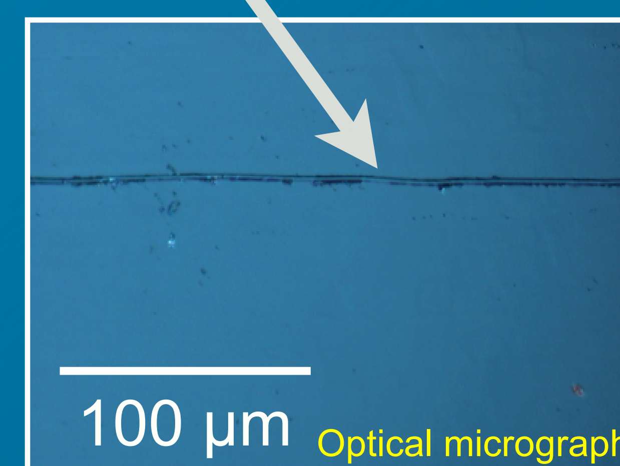
- Processing systems producing lengths of tape; R&D
- **Electropolishing**
  - polished > 2 km of tape
- **IBAD**
  - processed 100’s of meters of tape
  - continuous piece > 10 meters (FWHM ≤ 8°)
- **PLD**
  - processed 10’s of meters of tape
  - first continuously fabricated meter-long Research Park CC’s on IBAD-MgO
- Variety of samples sent to collaborators
- LA Research Park established as a User Facility



Electropolishing

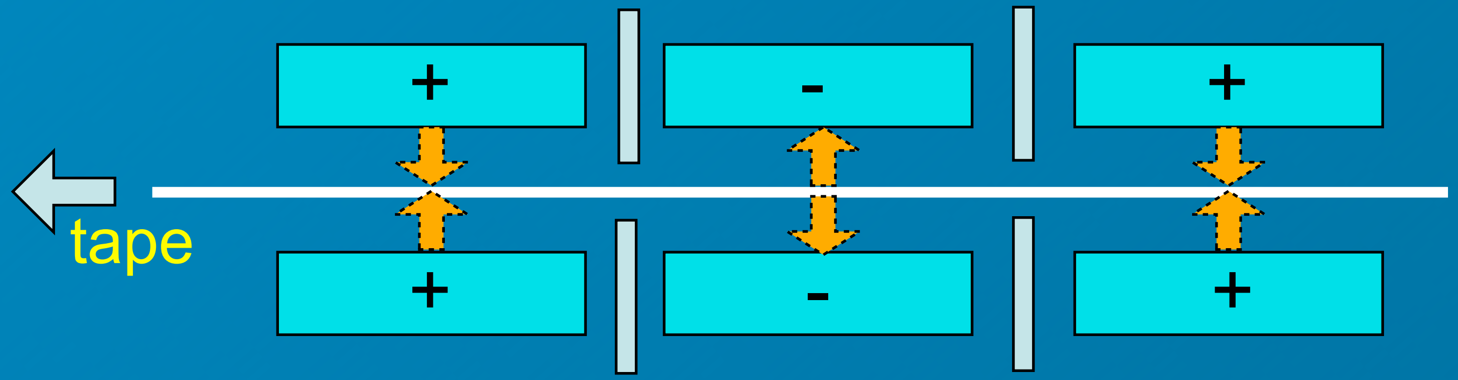
- Last year showed very good polishing results on Hastelloy C-276 with as-delivered system
- However, contact brushes and/or rollers often damage tape

Contact brushes can cause scratches



Bipolar setup for electropolishing

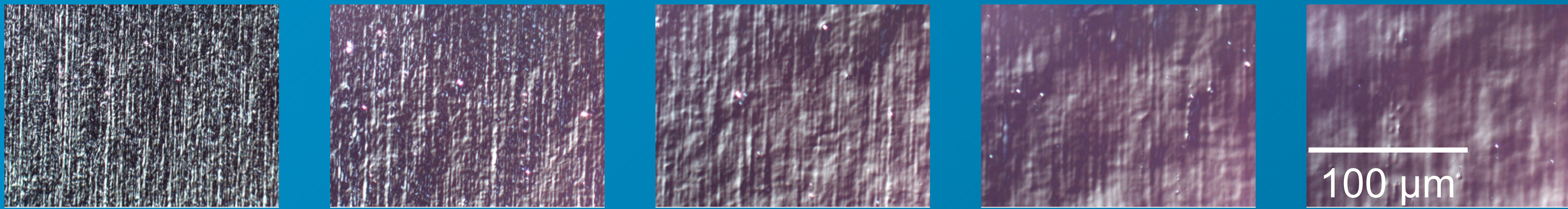
- Utilize electrolyte for electrical contacts to tape to avoid scratches



- Works very well for surface finish; better than physical contact

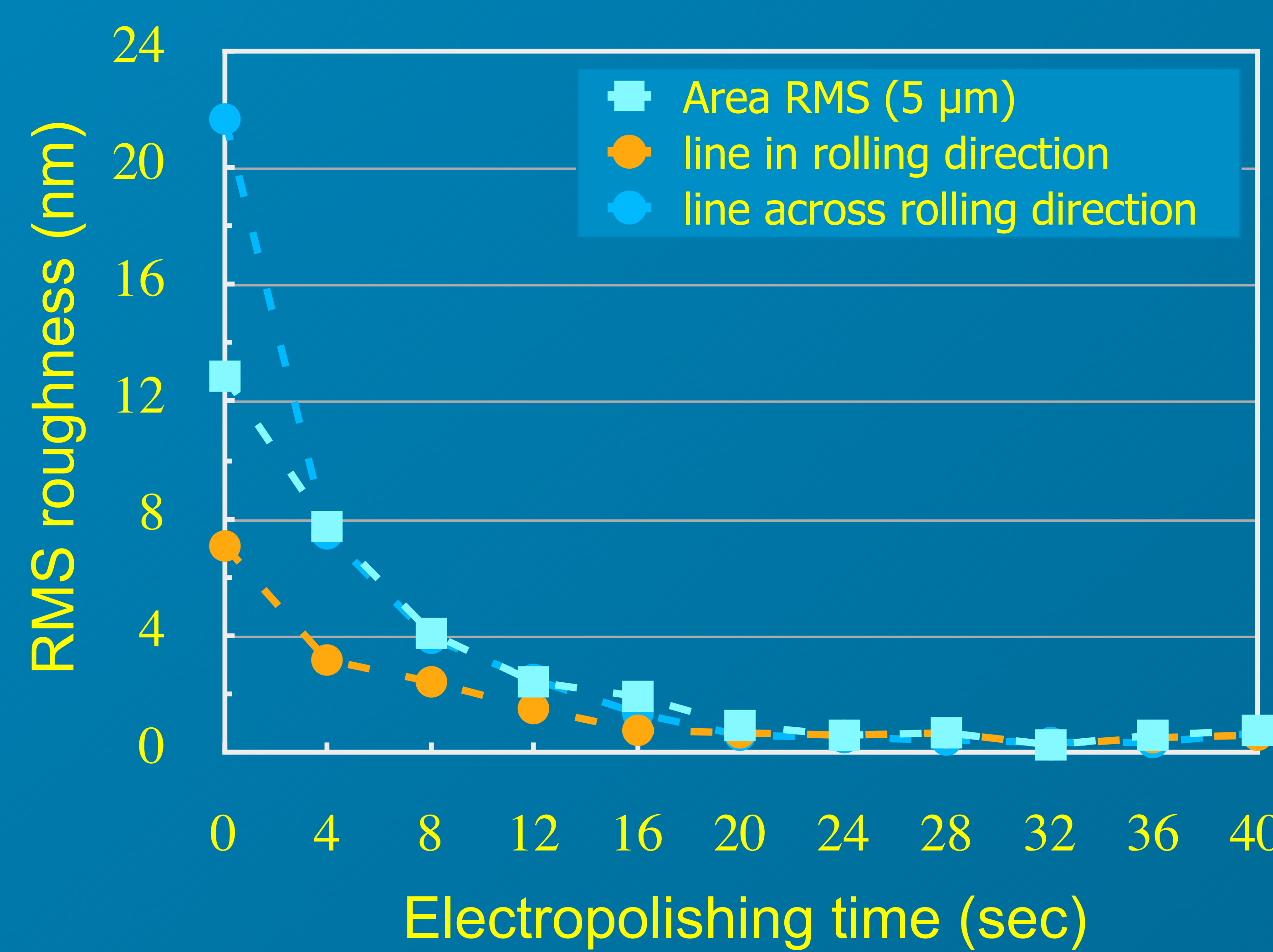
- Anodes have larger area than cathodes

Electropolishing vs time



4 sec | 12 sec | 20 sec | 28sec | 36 sec

- Results shown for 14 A
- Relatively wide process window
- RMS roughness can be < 0.5 nm on 5 x 5 µm

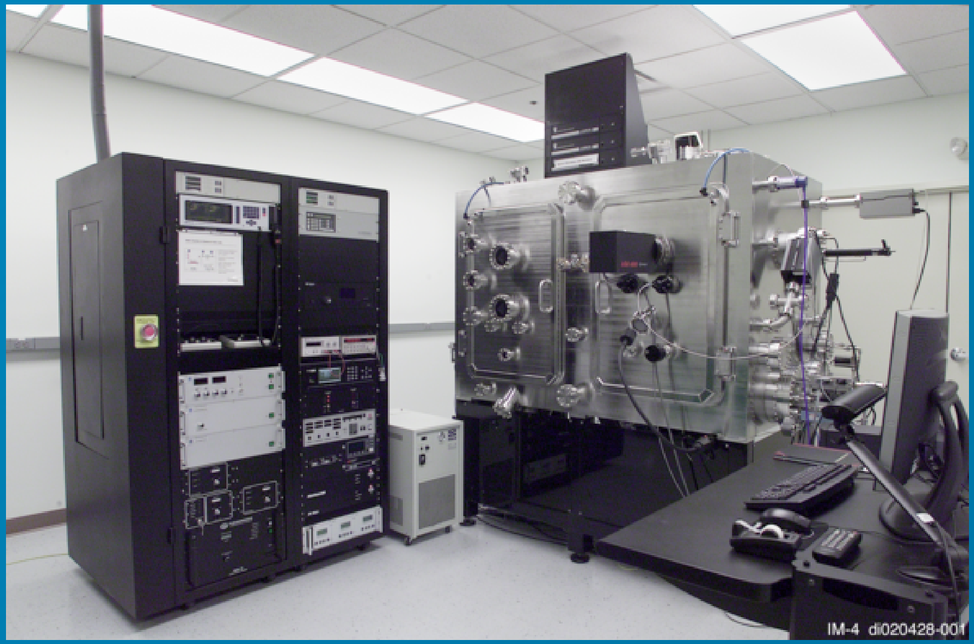


Speed of electropolishing

- Currently running electropolisher at 20 cm/minute or 12 m/hour, using two 10 cm-long cathodes.
- We extrapolate that these polishing results could also be achieved with wider tape, e.g. 10x, and with more electrodes, e.g. 8x.
- Our extrapolation yields **1 km/hour** of cm-equivalent tape.

IBAD

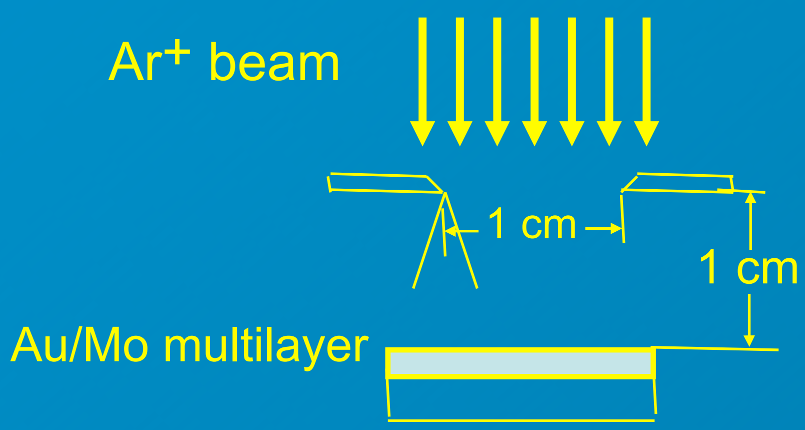
- Last year we showed results with 12° in-plane FWHM
- LANL Core Program was routinely getting 8° in-plane FWHM in MgO
- We wanted to understand what causes the "less good" 12° IBAD texture from RP



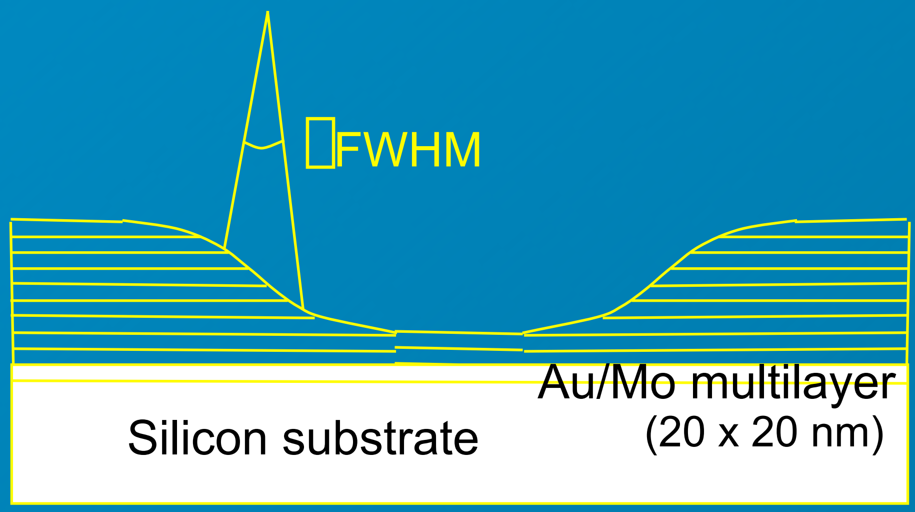
Issues that could affect IBAD texture:

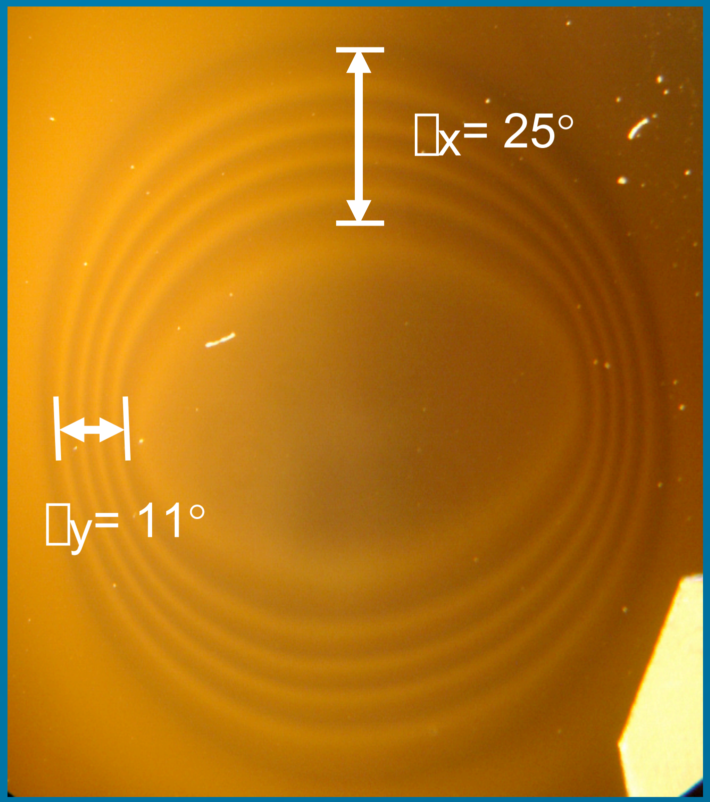
- Ion beam divergence
- Ion-to-molecule ratio
- IBAD layer thickness
- Nucleation layer

Measurement of ion beam divergence



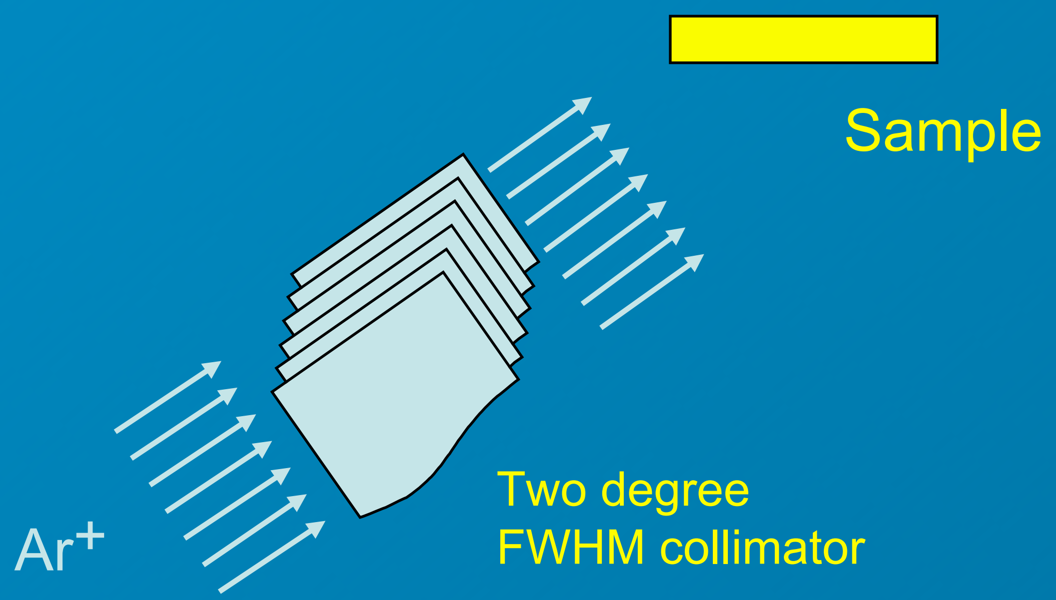
- Tool for measuring in-beam divergence
- Beam is broad with as-supplied source (~25°)
- This tool allows us to optimize divergence
- Ref: J. R. Kahn, H. R. Kaufman, C. A. Phillips, and R. S. Robinson, J. Vac. Sci. Technol. A **14**, 2106 (1996).

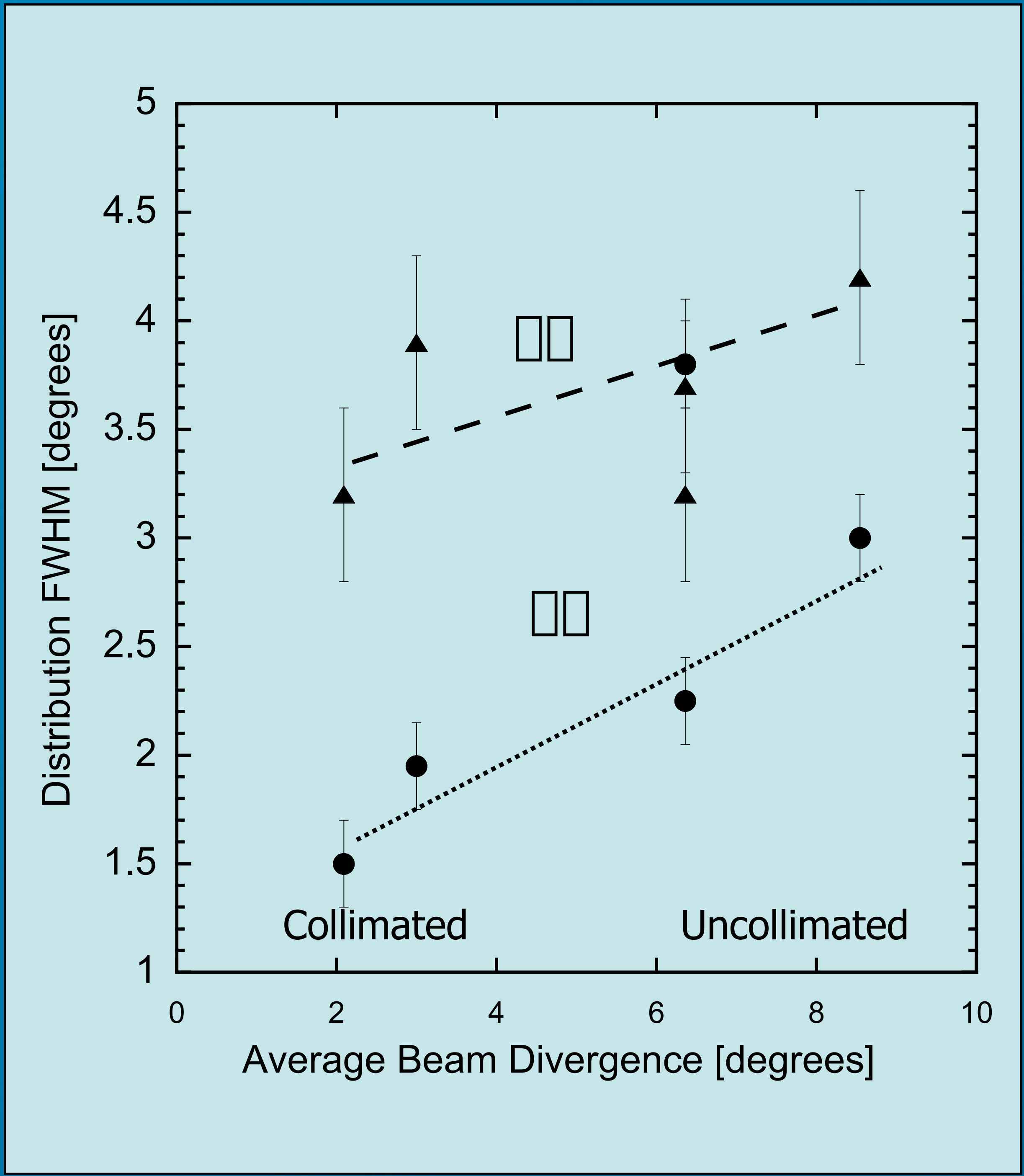




Texture Dependence on Beam Divergence

- Attempt to establish the limits of IBAD texturing
- How "good" can the IBAD layer be if we minimize the beam divergence

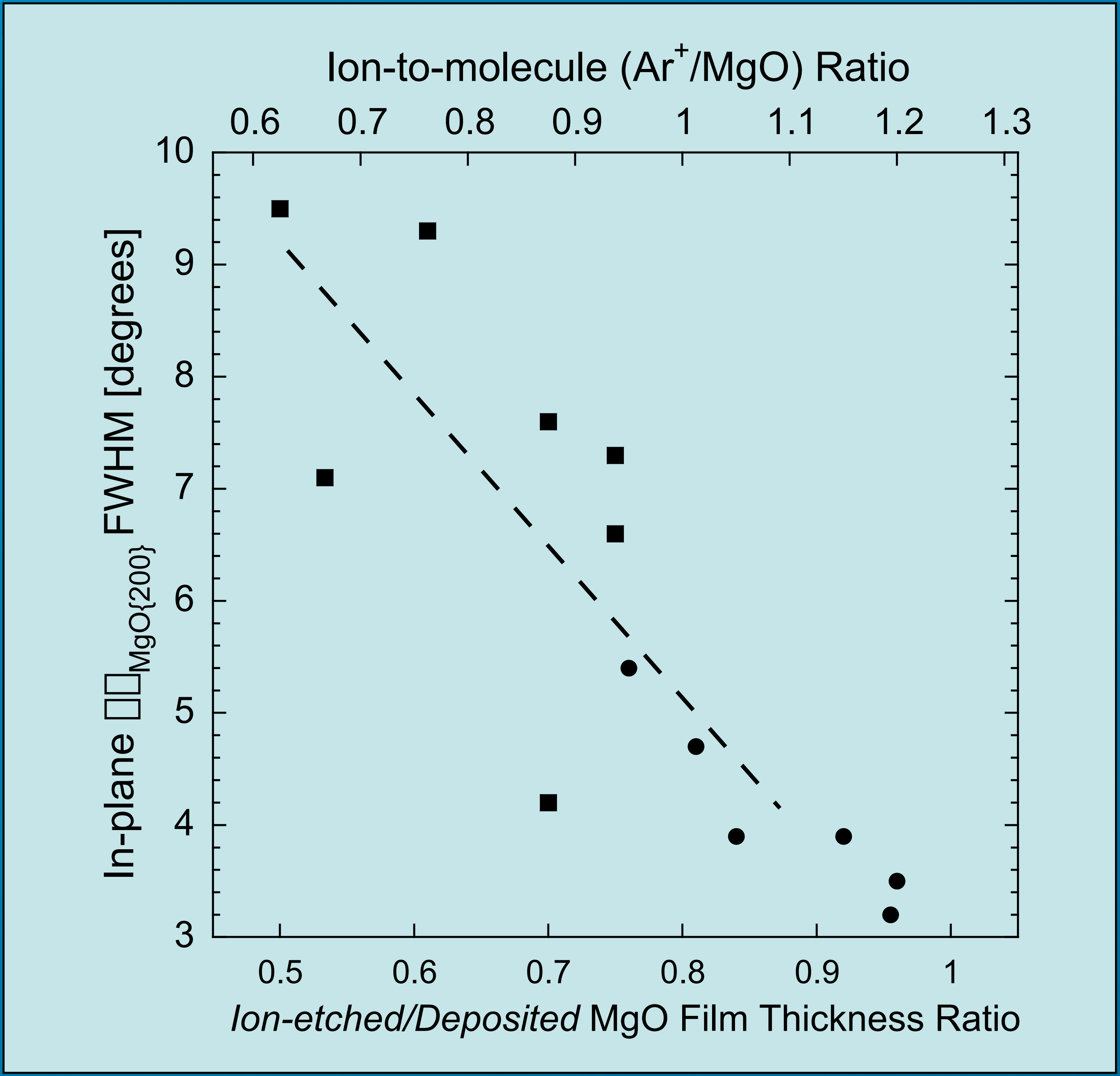




A. Findikoglu et al 2003, submitted for publication

Ion-to-molecule ratio

- Most **critical** parameter for IBAD-MgO formation and texture
- Accessible range of ratios depends on the nucleation surface



IBAD Process status

- IBAD process successfully transferred from Core Program to RP
- Producing 6-7° in-plane FWHM textured IBAD tape in longer lengths (> 5 m); 4-5° FWHM on tape without Al_2O_3 barrier layer
- We believe we understand the key parameters to get well-textured IBAD

Do we now understand everything?

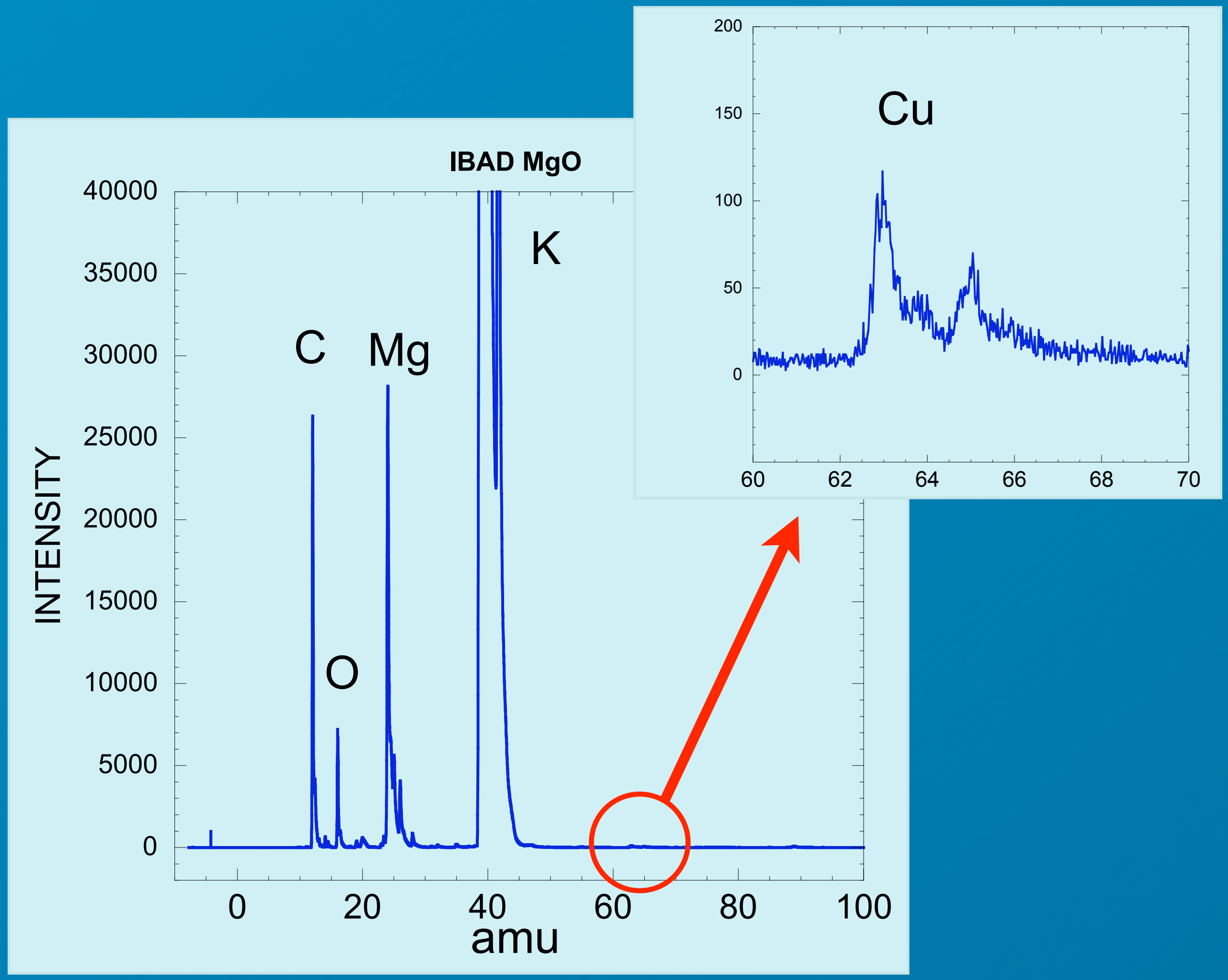
NO! But we are narrowing down the parameter phase space.

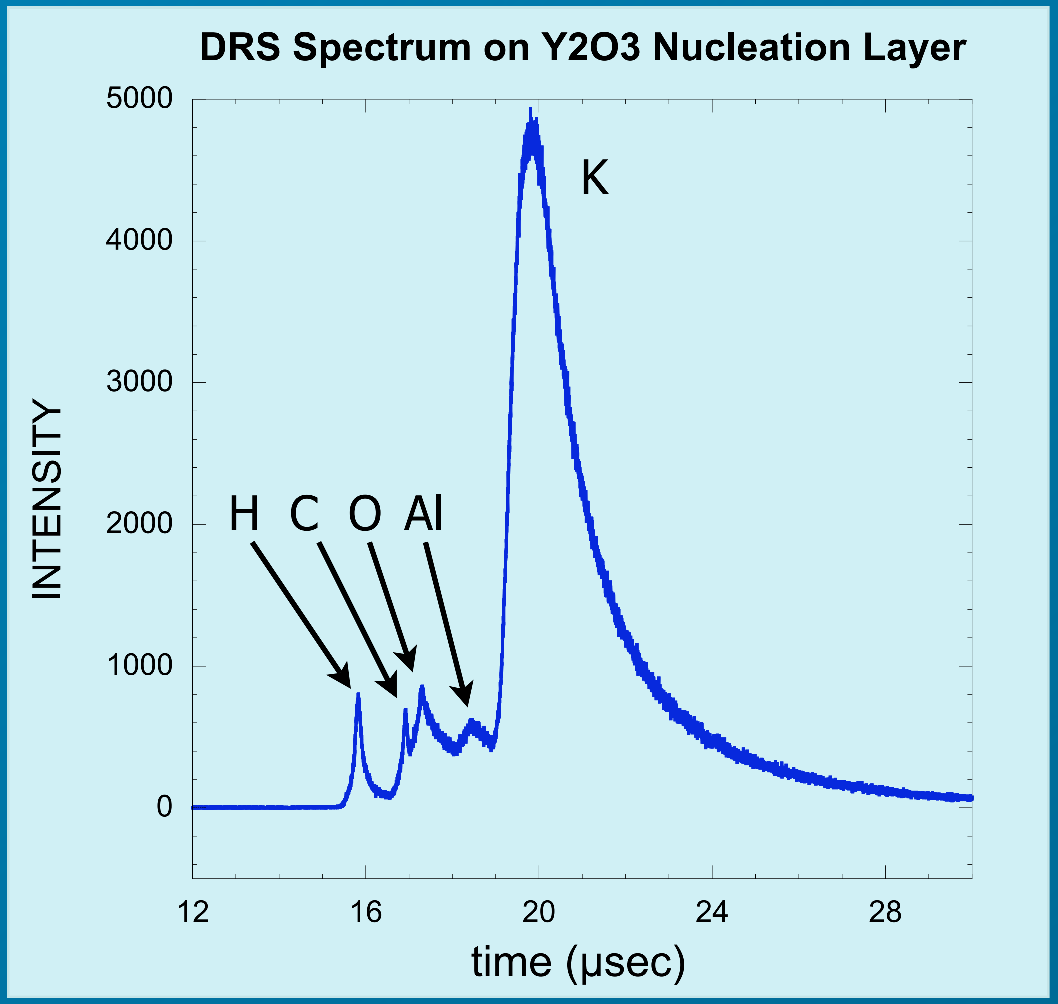
Surface control and the right nucleation layer are key.

We believe *in situ* diagnostics are very helpful. (RHEED)

Time-of-Flight Ion Scattering (TOF-ISARS)

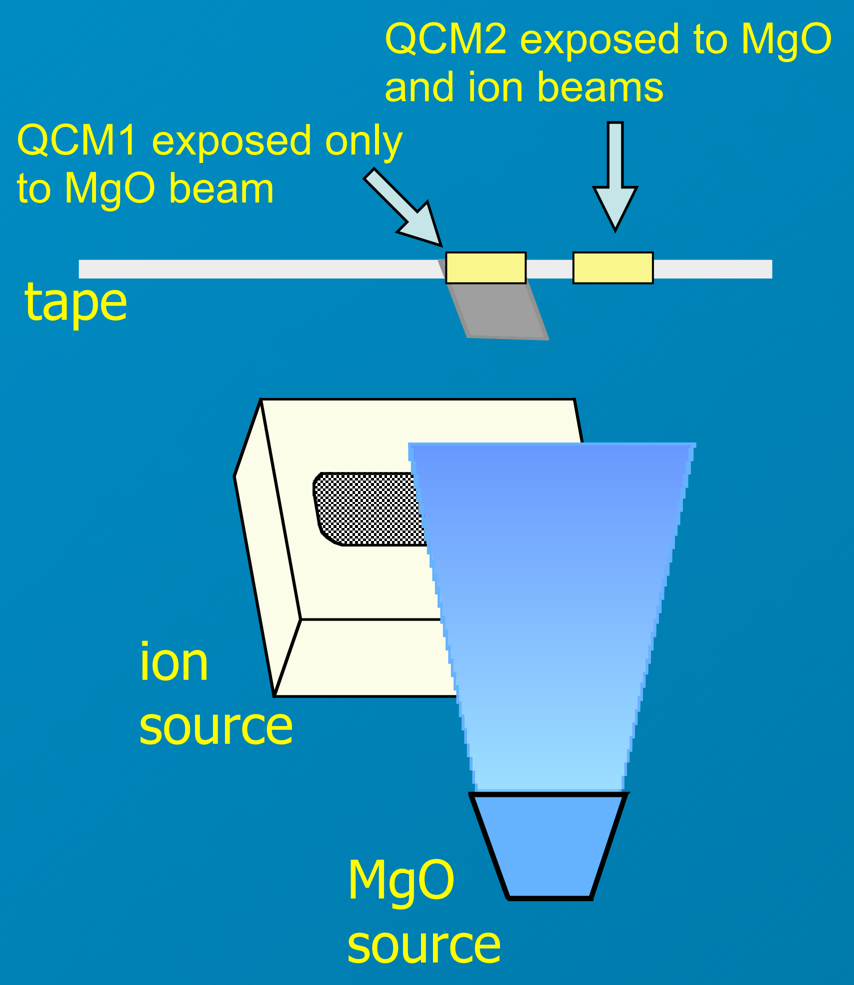
- Implementing in our IBAD system for *in situ* surface characterization
- IBAD-MgO samples sent to Ionwerks for initial characterization
- Surface analysis shows small amounts (≤ 1%) of Cu on the surfaces
- Al from Al_2O_3 is seen on the surface of a heated Y_2O_3 nucleation layer

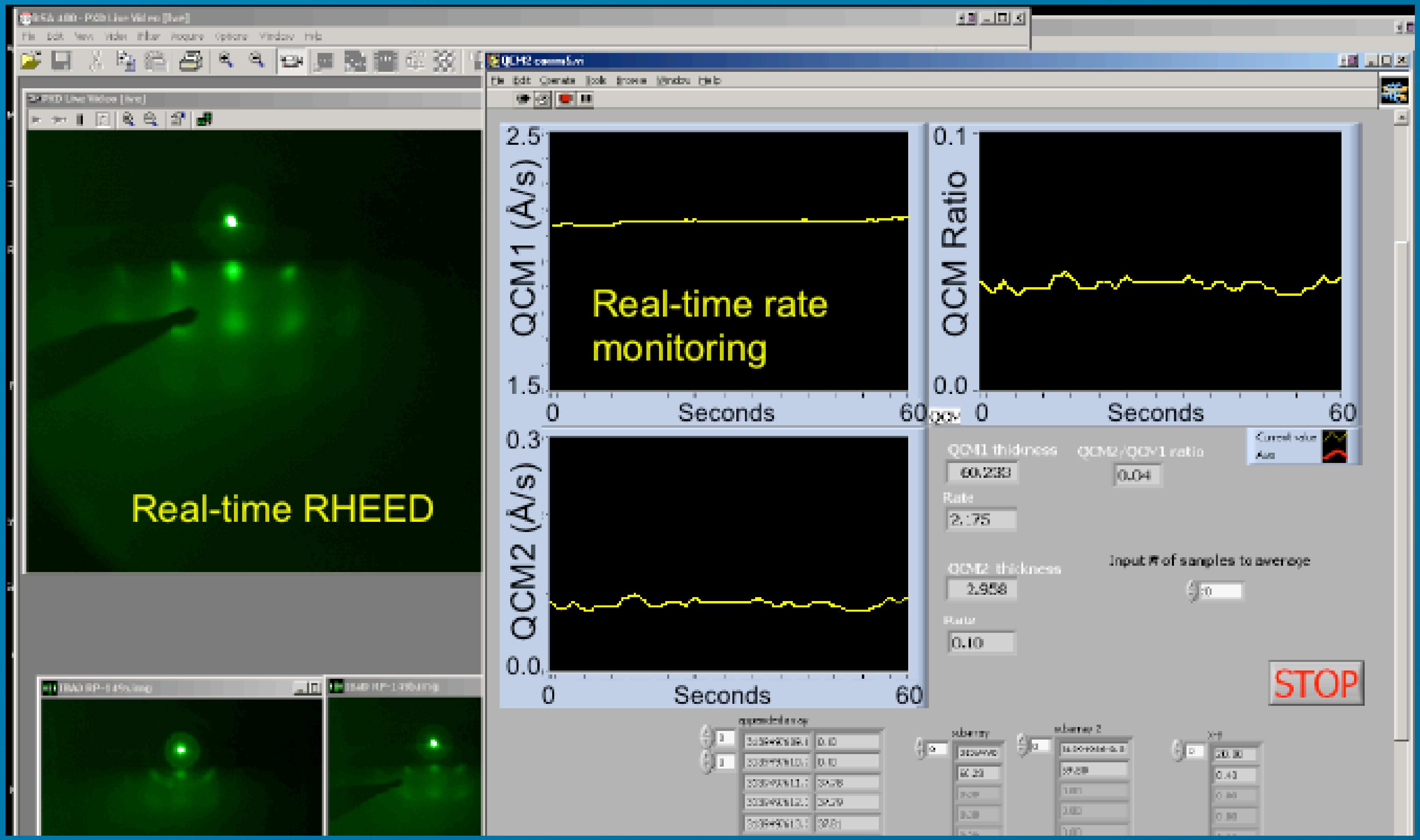




IBAD Process Control

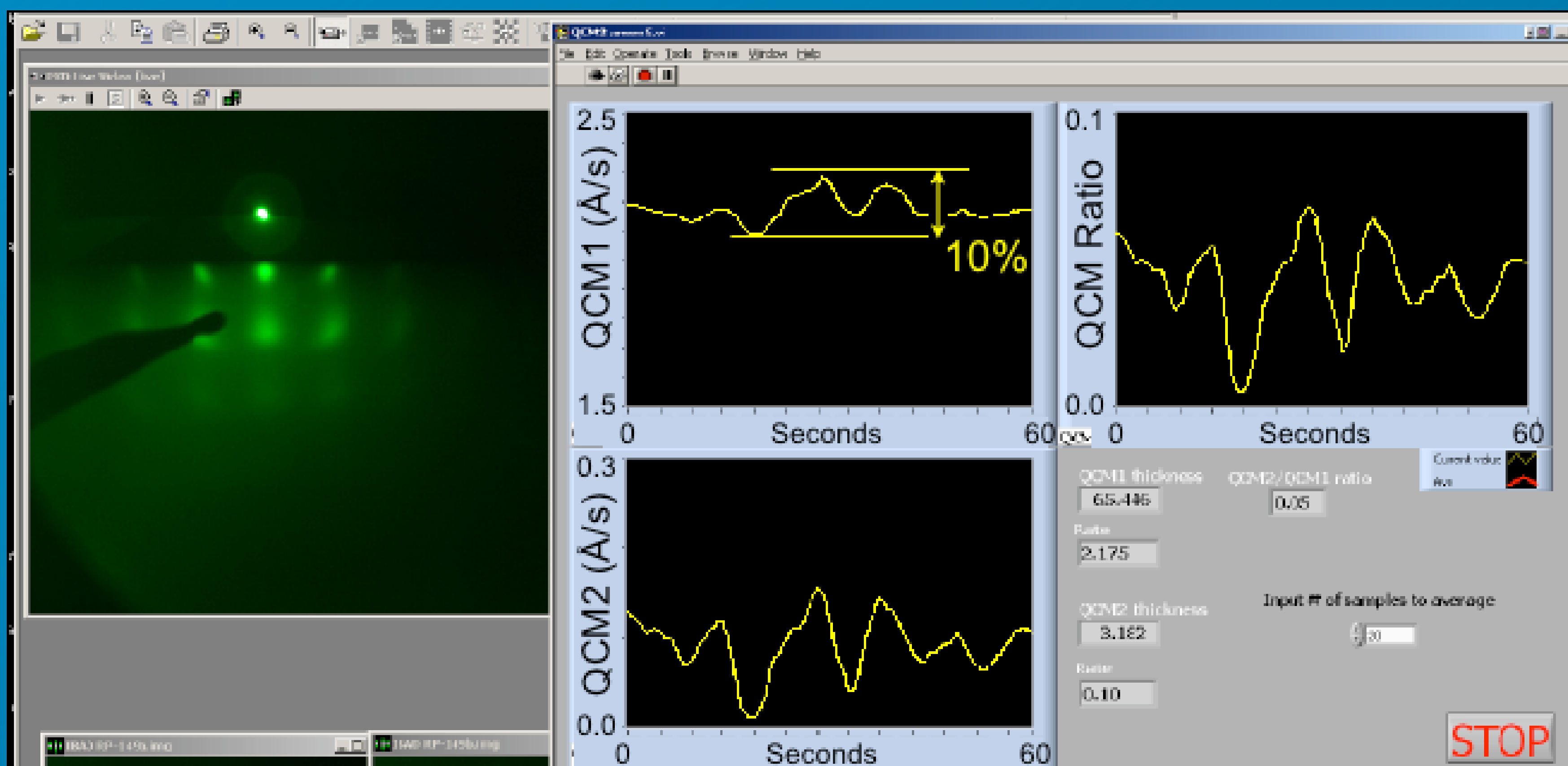
- Utilize 2 quartz crystal monitors for measuring IBAD deposition rates
- One measures only MgO deposition and the second measures etched (deposition - etching) rates
- Can achieve stable deposition over > 10 meters



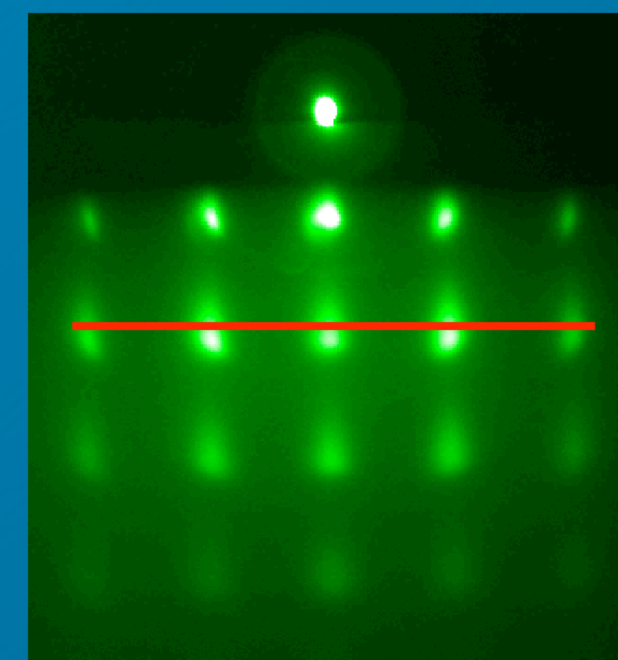


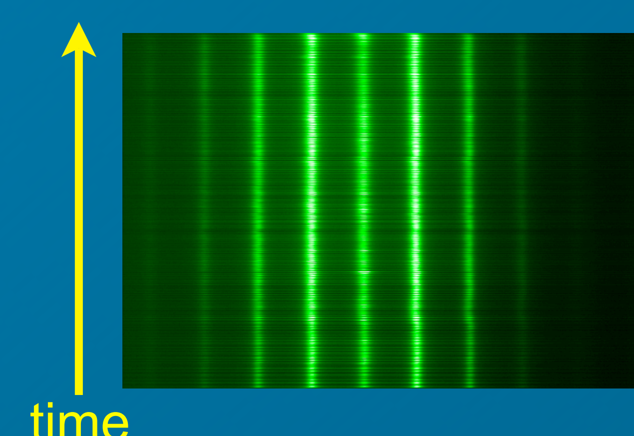
IBAD Process Control - cont'd

IBAD process tolerates ± 5% rate variation with less than 0.5° texture difference



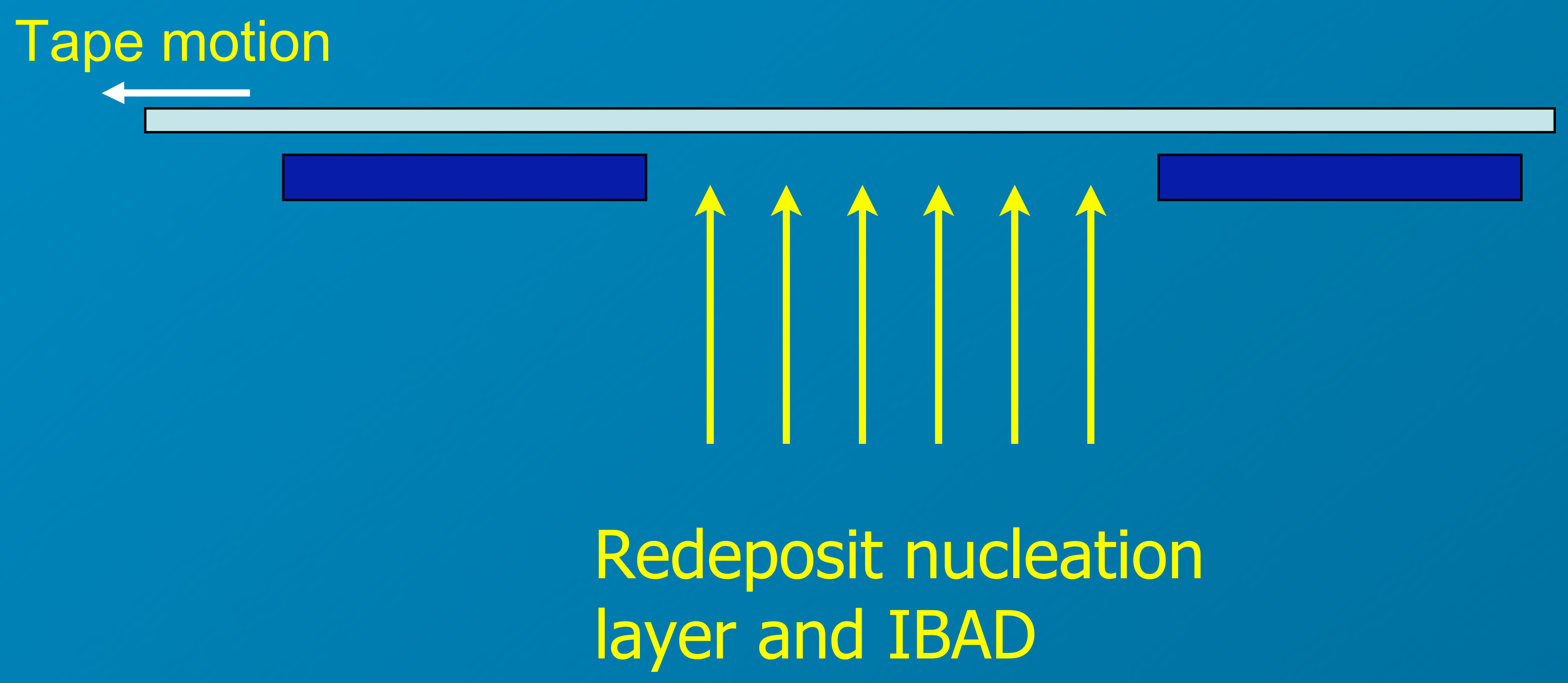
RHEED of 10 meters of IBAD after epi-MgO deposition (QC movie)





Reworkability

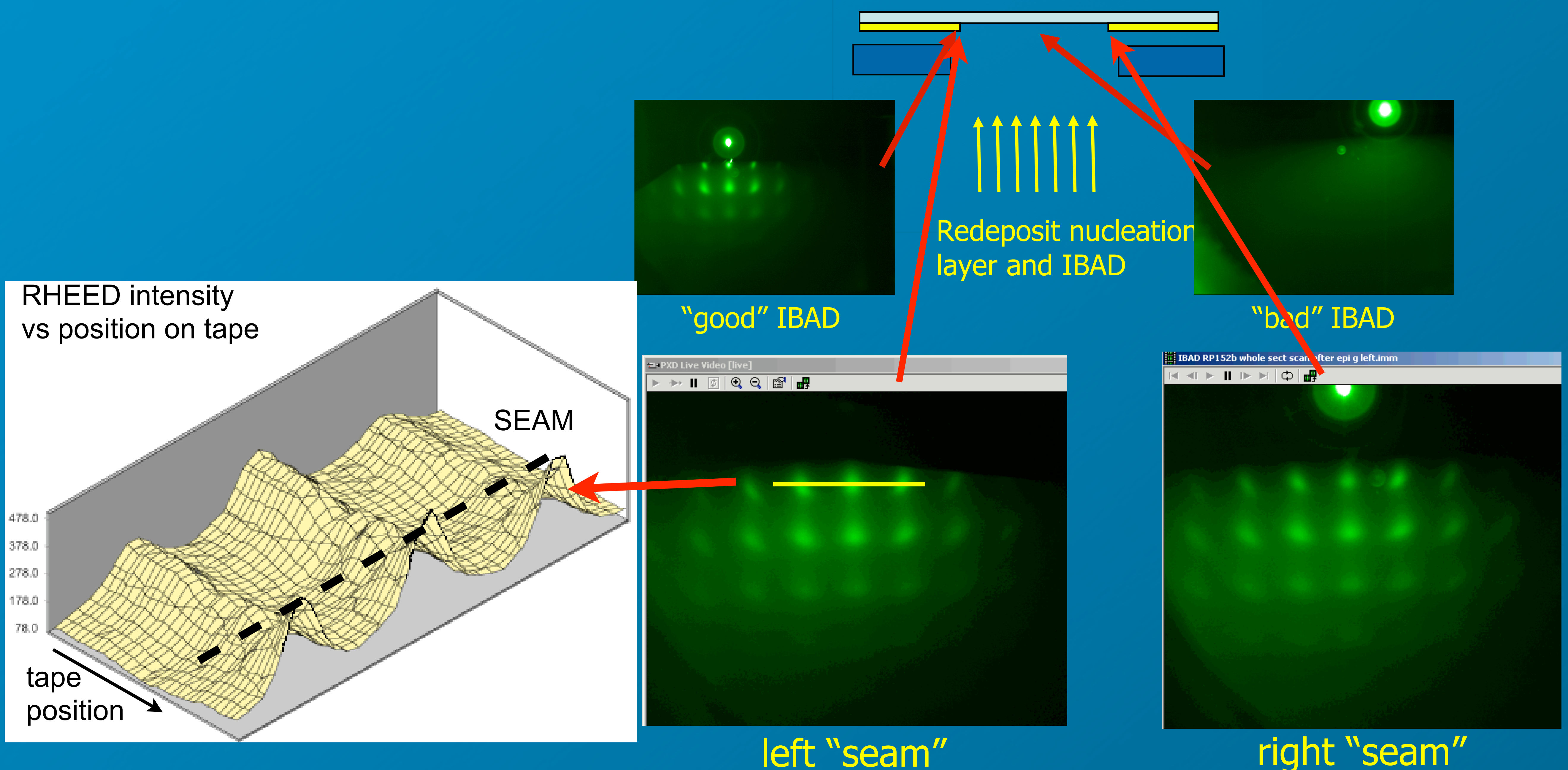
- Known for long time that IBAD tape can be reworked
  - MgO can be etched away and new nucleation layer and IBAD layer deposited



Reworkability - Repair

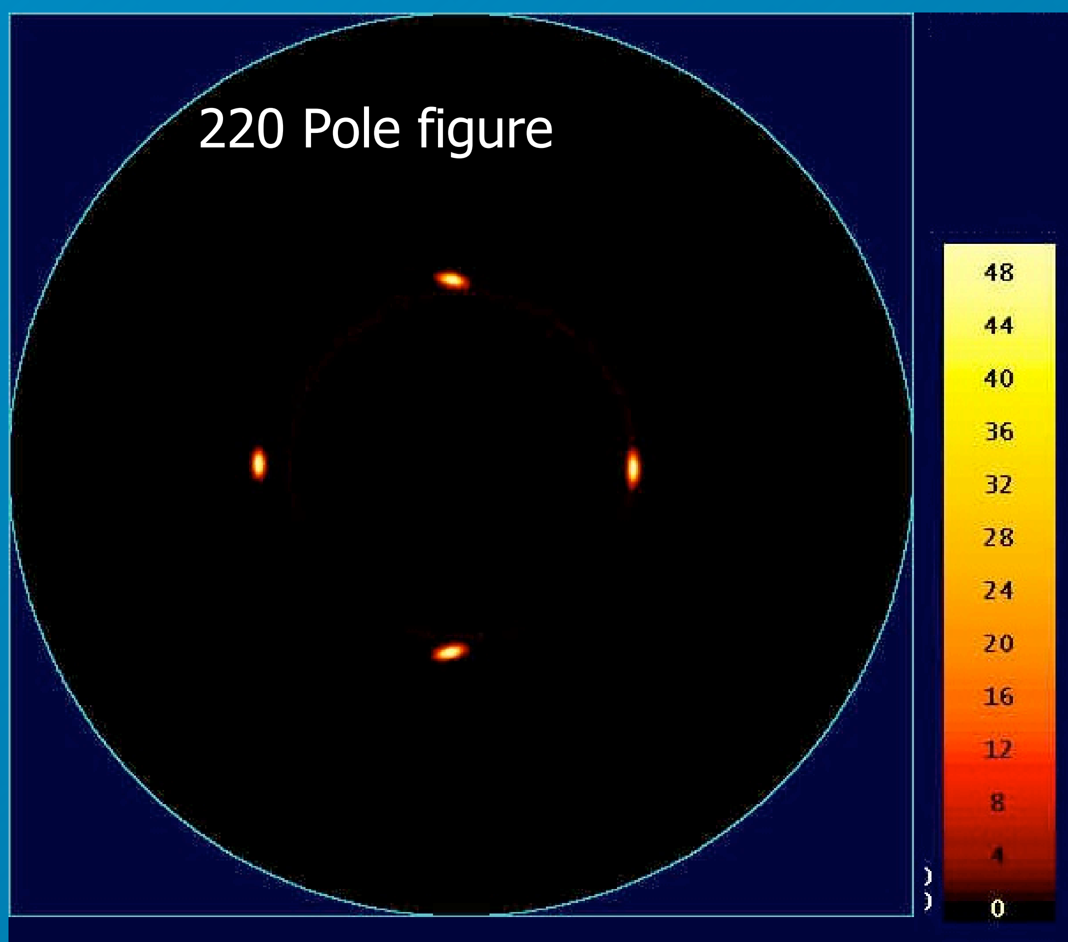
Can we repair a bad IBAD section?

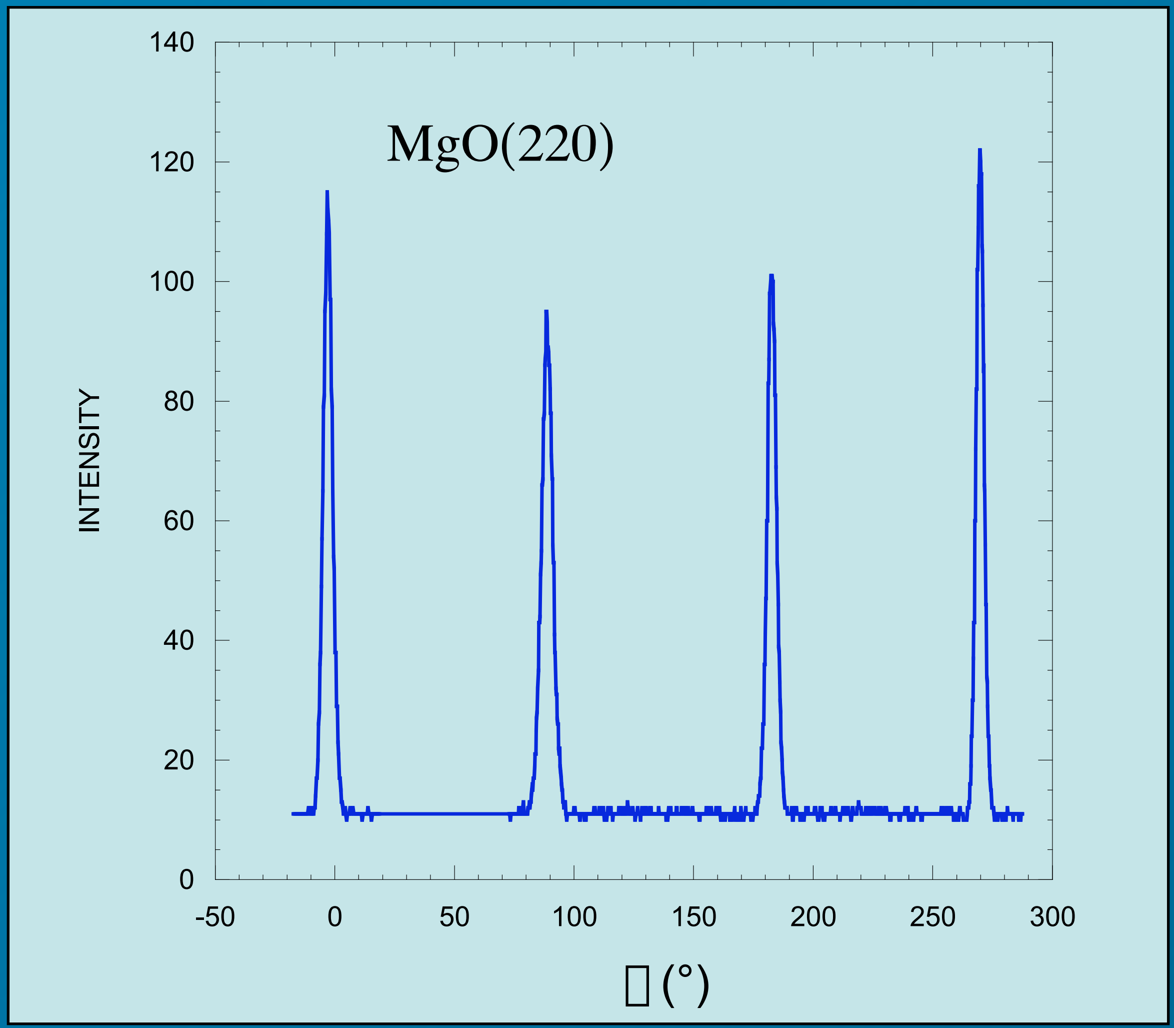
Demonstrated repair in RHEED "proof of principle"



High Rate IBAD-MgO Process

- Normal tape speed for RP is 5 - 10 m/hour (~2 mm/sec)
- To test how scalable IBAD-MgO is we increased the ion current to the source maximum (~ 320 mA)
- Adjusted the MgO rate and tape speed accordingly (5x)
- Result: FWHM in-plane 4.5°, out-of-plane 1.4°





Speed of IBAD-MgO Process

- We have demonstrated 36 meters/hour with 4.5° in-plane FWHM
  - Speed limited by the ion gun utilized
- We can extrapolate to 50% longer deposition zone and 5x wider tape yielding 250 m/hour of cm-equivalent tape (with same ion gun)
- Four ion guns could be placed in series to increase the throughput to **1 km/hour** of cm-equivalent tape

IBAD-MgO Summary

Excellent texture

Fast process

Robust

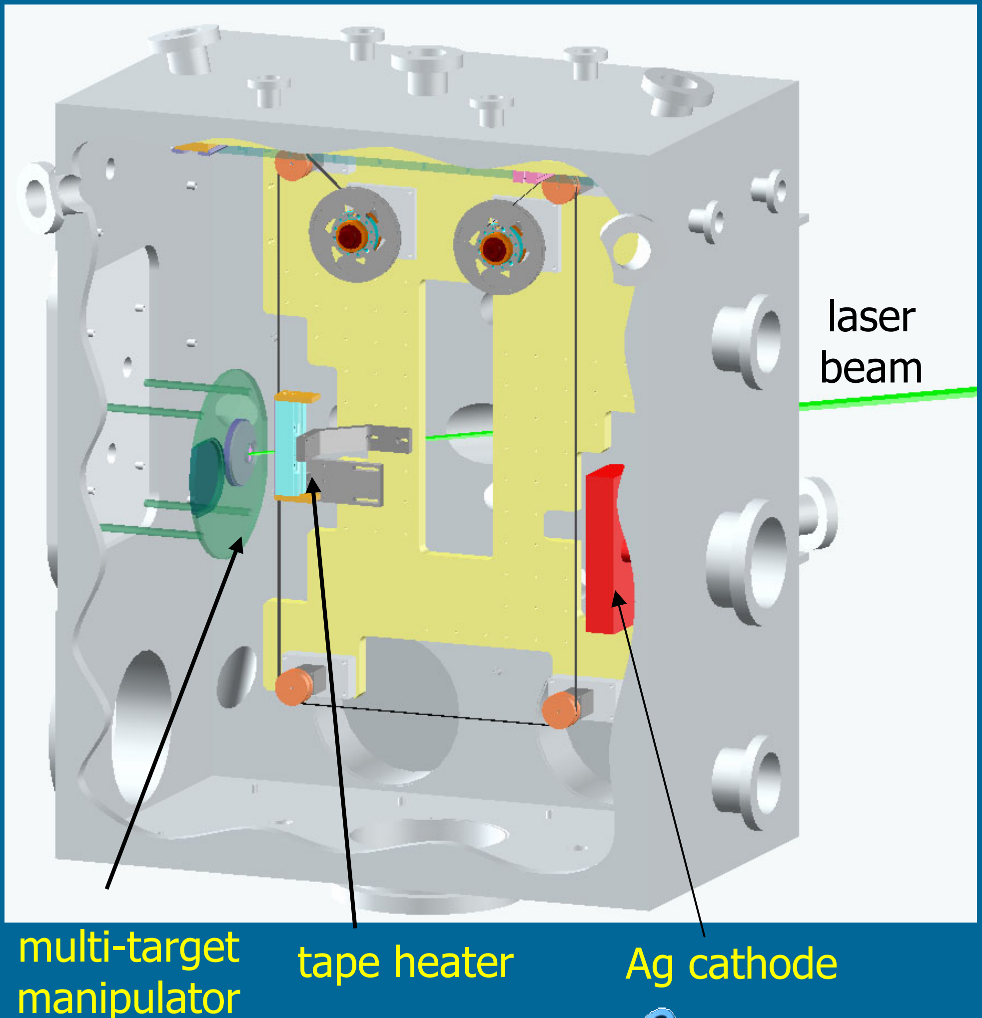
Long length capable

Flexible (for smooth surfaces)

Reworkable (repair)

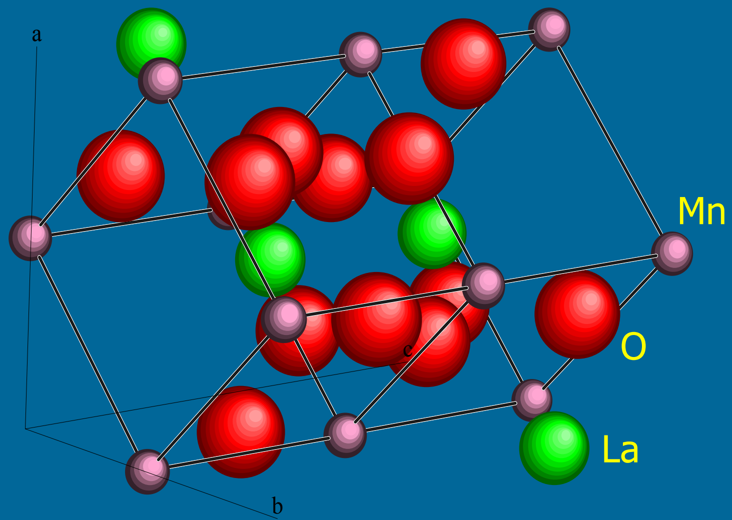
Reel-to-Reel Pulsed Laser Deposition Chamber

- 200 W XeCl (308 nm) excimer laser – 300 Hz @ 650 mJ
- Four 4″ targets for deposition of a variety of oxide layers
- Lengths from 1 cm to 100’s of meters
- Quartz lamp heater heats tape as it continuously moves through the PLD zone
- *In situ* adjustment of tape position with respect to laser plume
- Silver deposition integrated

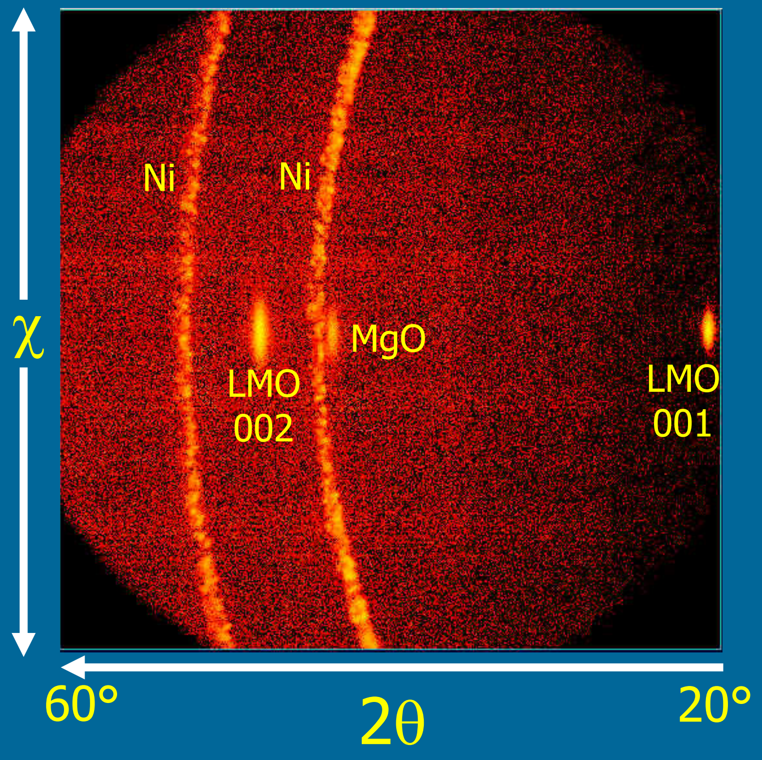


Buffer Layer

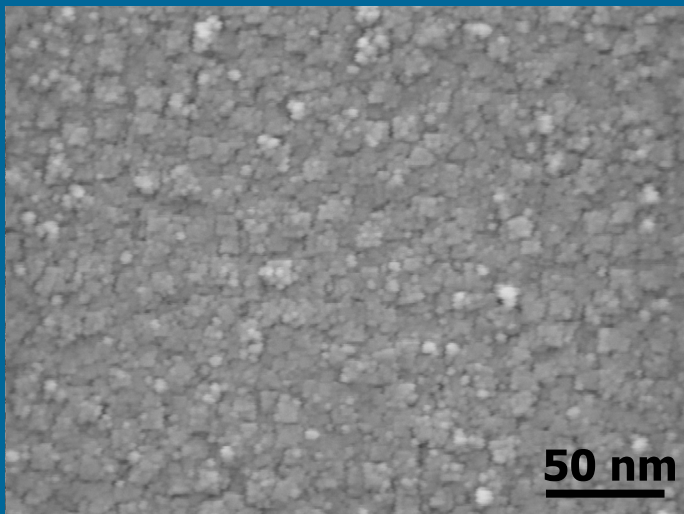
- $LaMnO_3$ buffer layer
  - Pseudo-cubic perovskite (110 spacing = 0.3985 nm)
  - Low deposition temperature
  - Wide temperature window
  - High deposition rate (0.1 nm/shot)
  - Very dense targets
  - Up to 21 m/hr (100 nm thick)



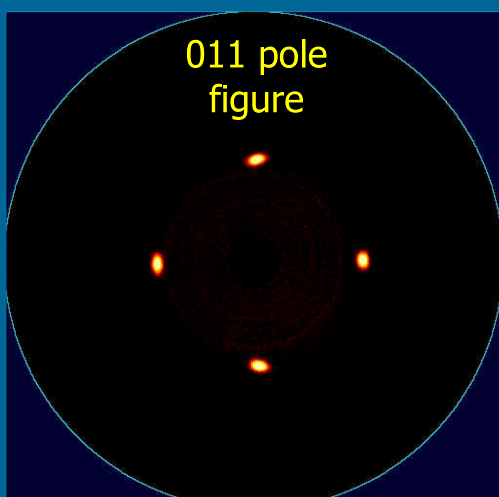
Bruker GADDS system θ-2θ frame

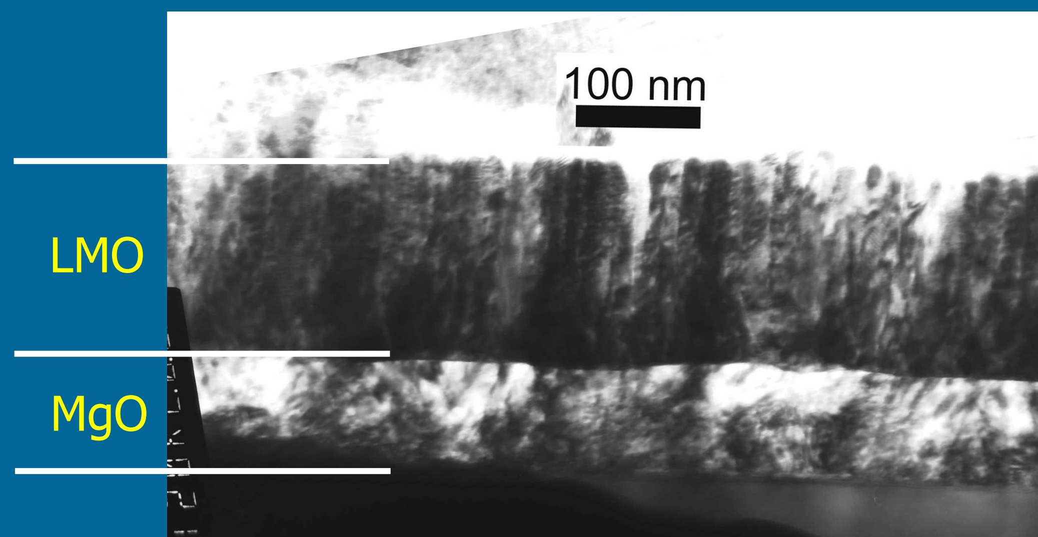


Buffer Layer



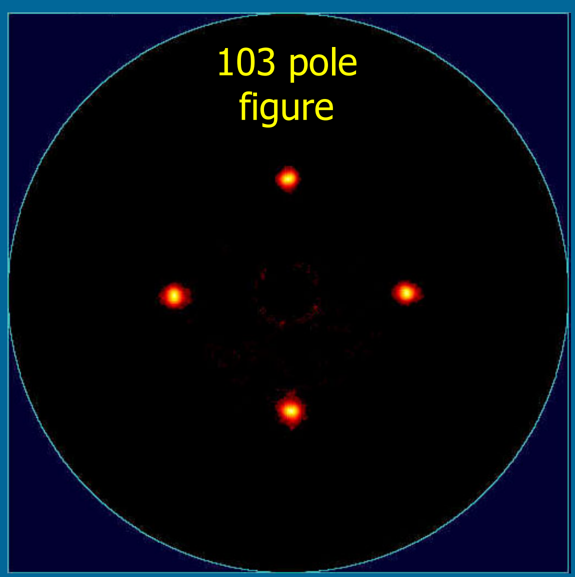
- Clean interface w/ epi-MgO
- Columnar microstructure
- Smooth surface (grain size ~ 10 nm)
- FWHM in ϕ improved over MgO by ~ 1 - 2°



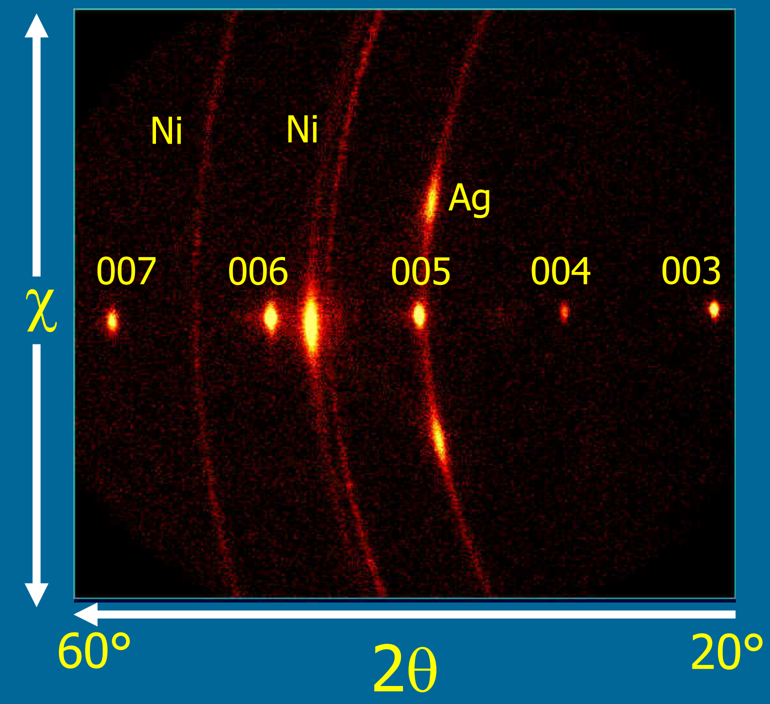


YBCO Deposition

- Using a 12 in. quartz lamp heater
- Quartz lamp temperature typically ~ 900 - 1000 °C
- Laser frequency up to 80 hz @ 650 mJ @ 200 mTorr
- Up to 5 m lengths

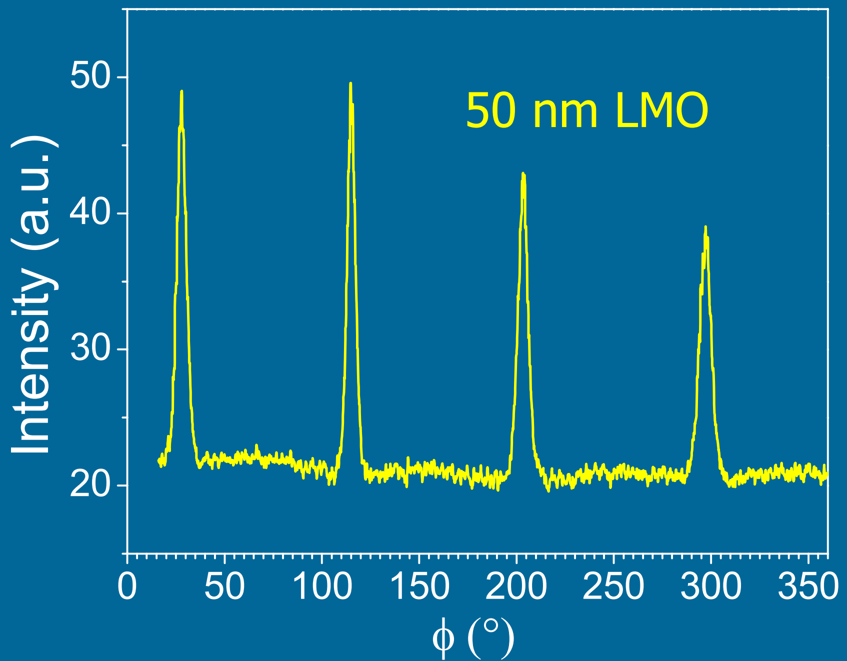


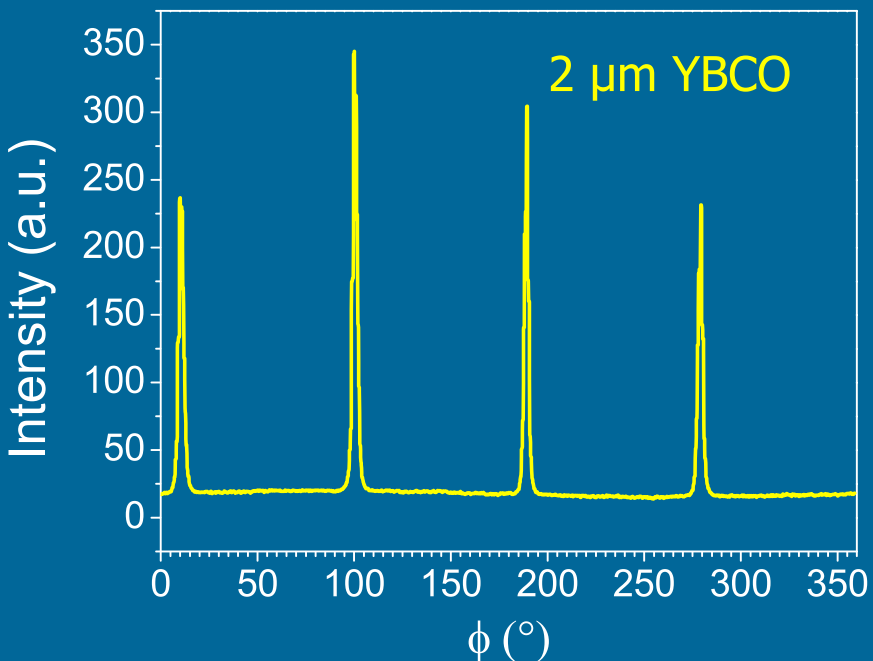
0.5 µm Ag/2 µm YBCO



Texture Development

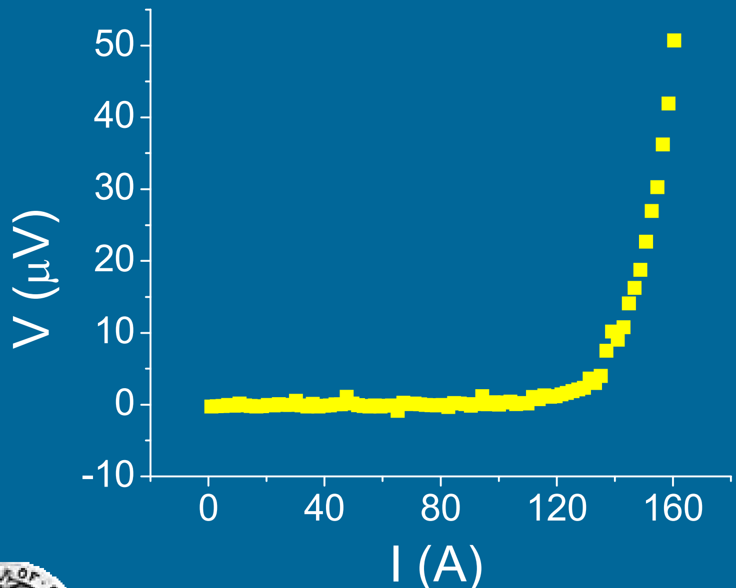
- Epi-MgO/IBAD-MgO/Hastelloy C-276 ϕ-FWHM ~ 6 - 7°
- 50 nm LMO ϕ-FWHM ~ 5 - 6°
- 2 µm YBCO ϕ-FWHM ~ 2.5 – 3.5°

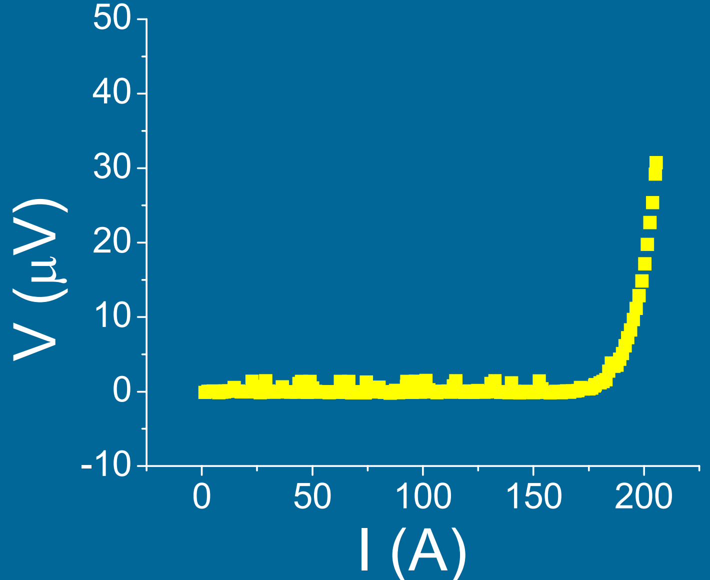




I_c Data

- 2 µm YBCO/50 nm LMO/Epi-MgO/IBAD-MgO/Nickel alloy
  - 178 A across 1 cm
  - 120 A across 10 - 20 cm
  - 50 A across 1.1 m
  - Microbridges - 1.1 MA/cm^2





Self field
75.5 K

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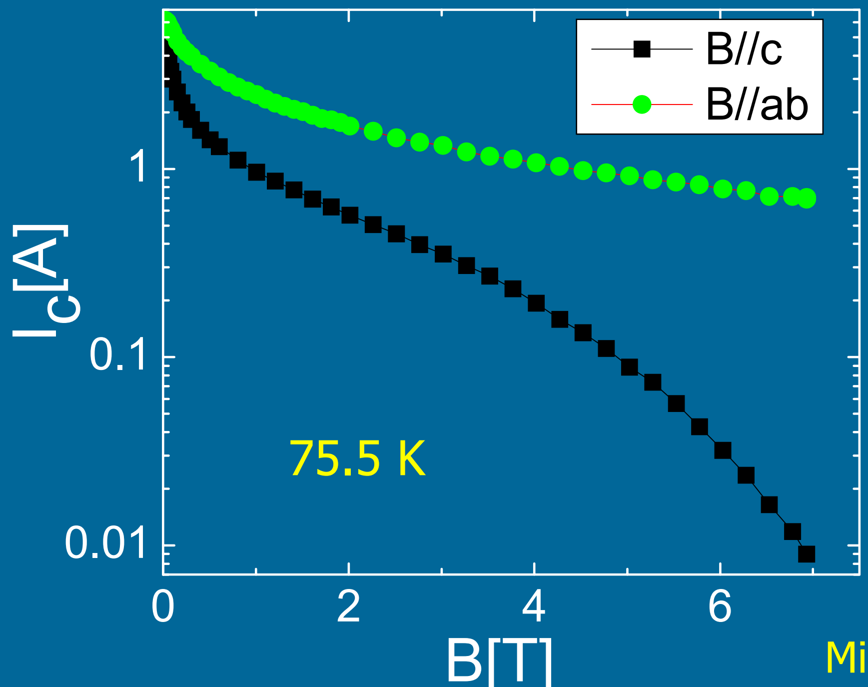
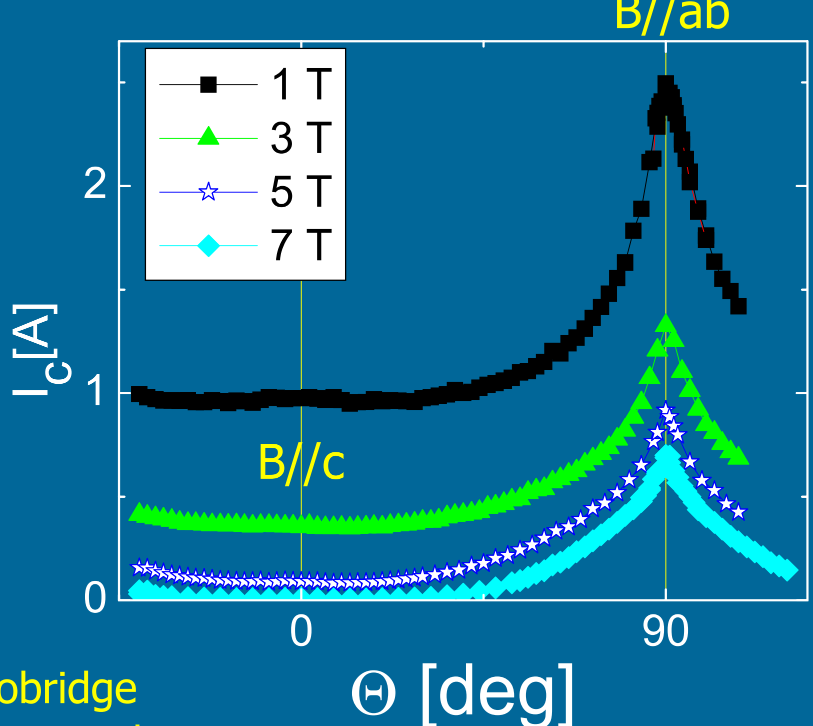
Field Dependence

- I_c vs. field data
  - Typical of PLD films
  - Evidence of a small peak in the c-axis direction



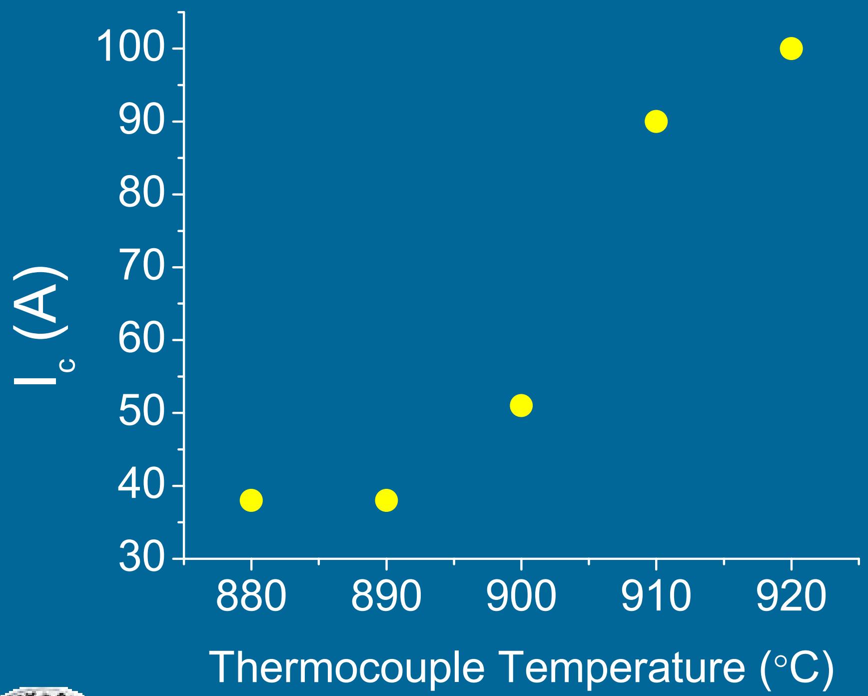


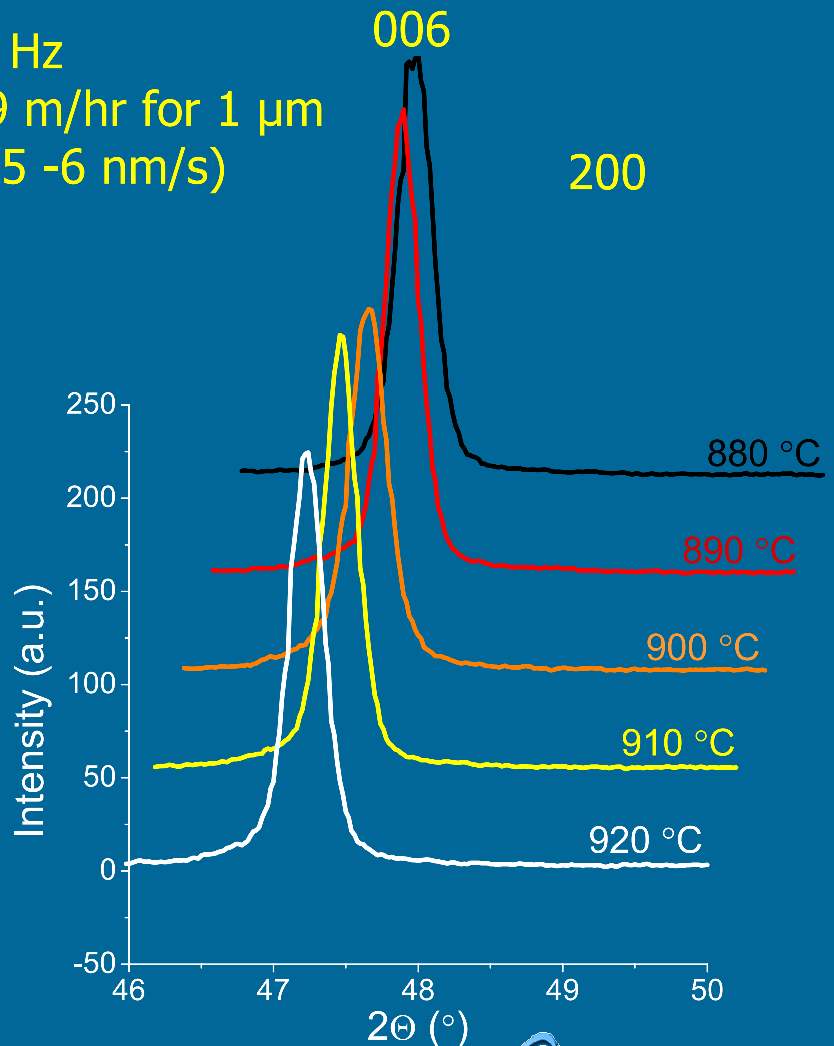
Microbridge measurements

Key Issues for Higher Deposition Rates

- As the rate increases, so must the temperature
- The window for quality films is smaller at higher rates

30 Hz
0.9 m/hr for 1 μm
(4.5 -6 nm/s)

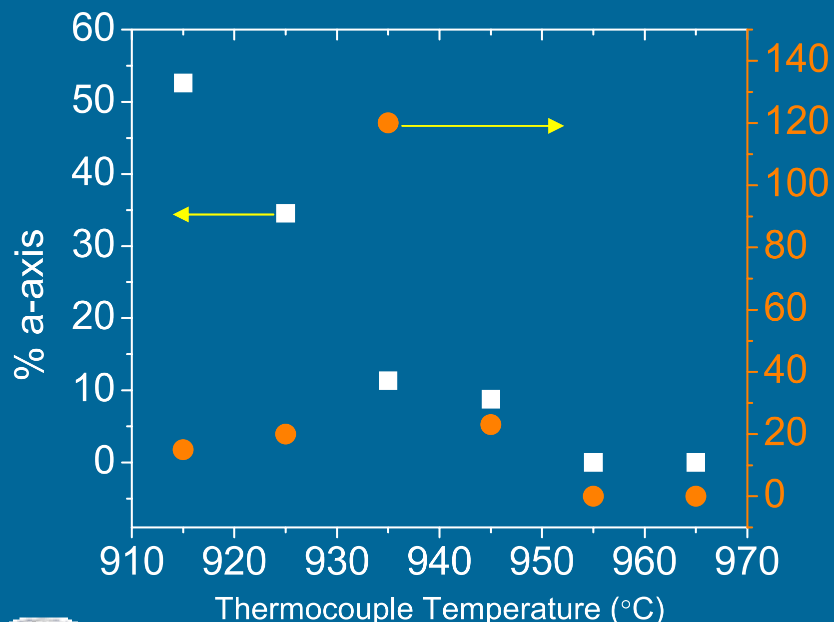


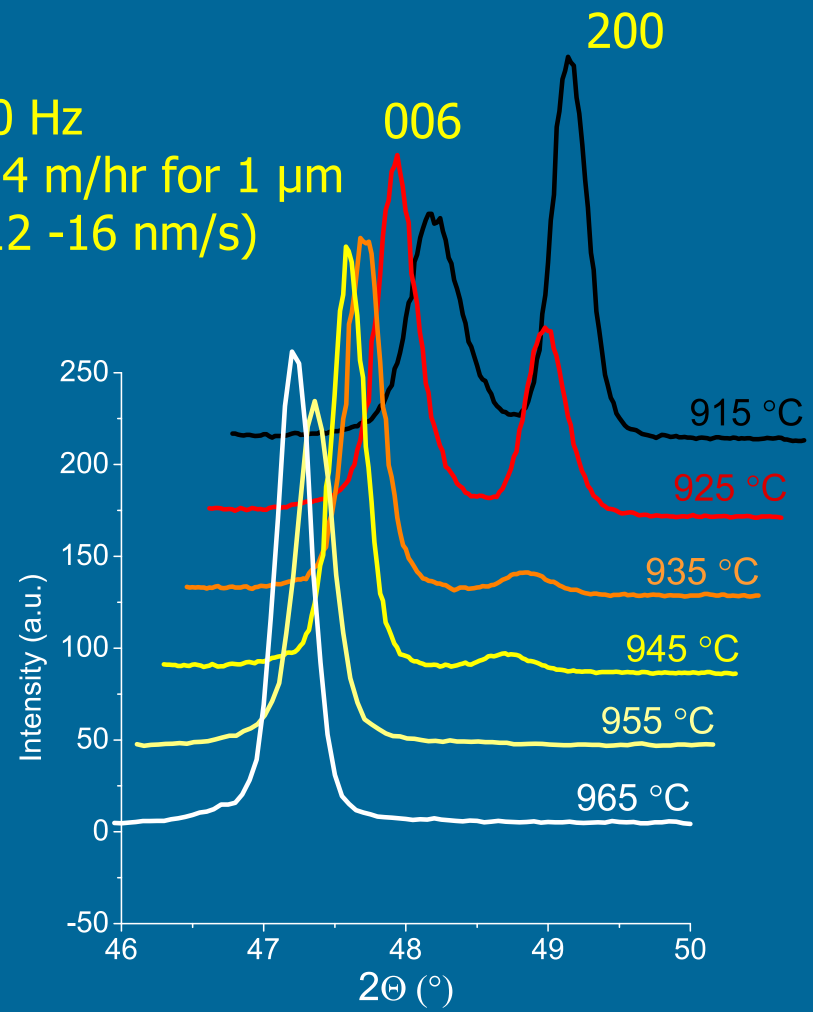


Key Issues for Higher Deposition Rates

- Temperature
  - Can we get hot enough for even higher rates?
  - Will the thickness between the YBCO and substrate have a minimum?

80 Hz
2.4 m/hr for 1 μm
(12 -16 nm/s)





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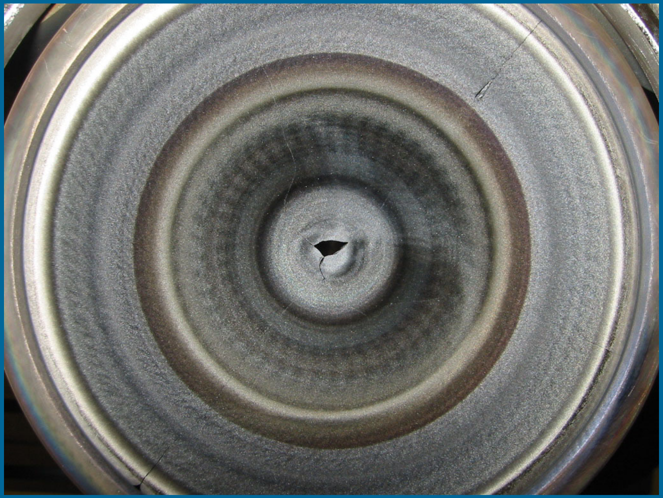
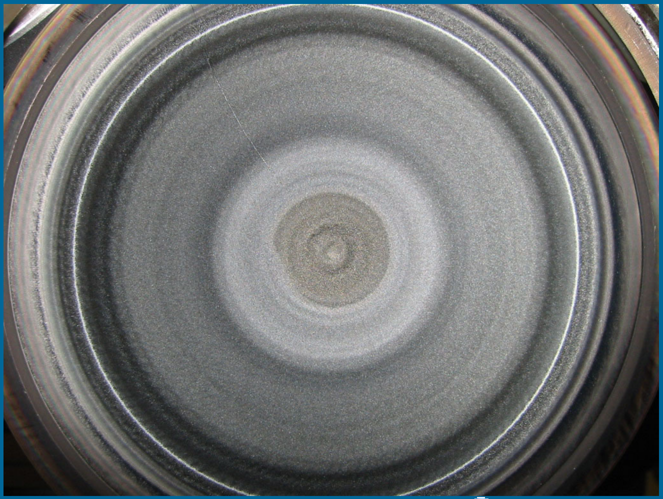
Key Issues for Higher Deposition Rates

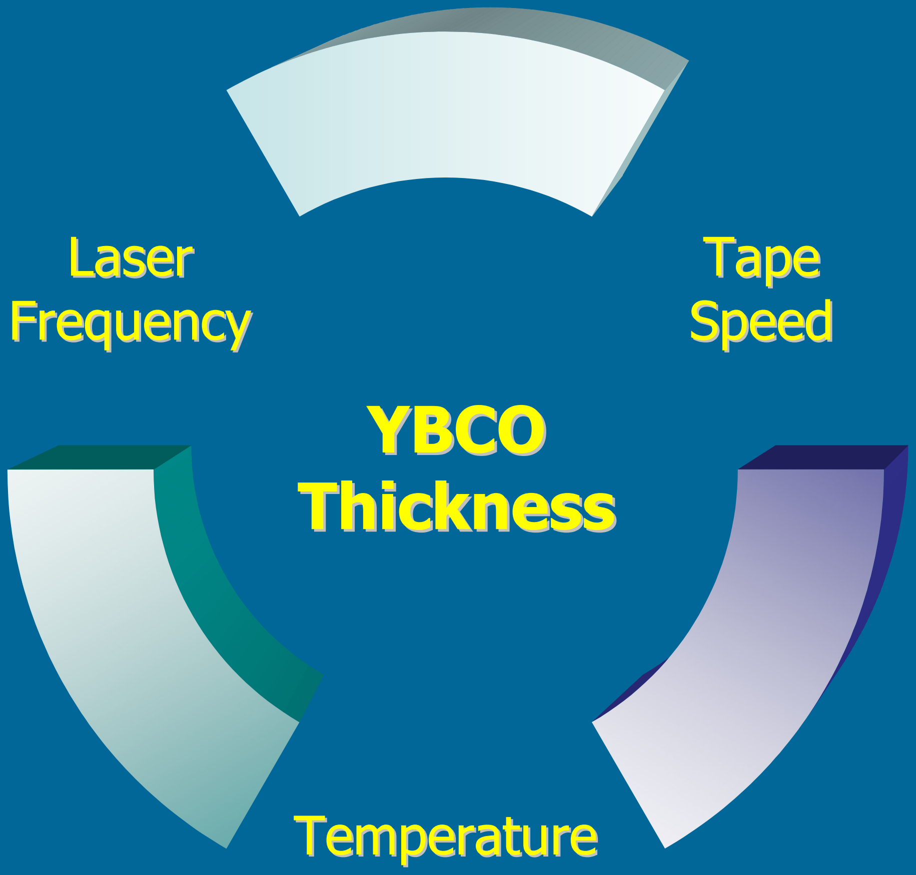
- As we approach the limits of the laser (300 Hz), plume stability and uniformity becomes even more important
- Target rastering
  - To get maximum utilization
  - To minimize surface modification/plume tilting
    - Need precise control over target position/velocity





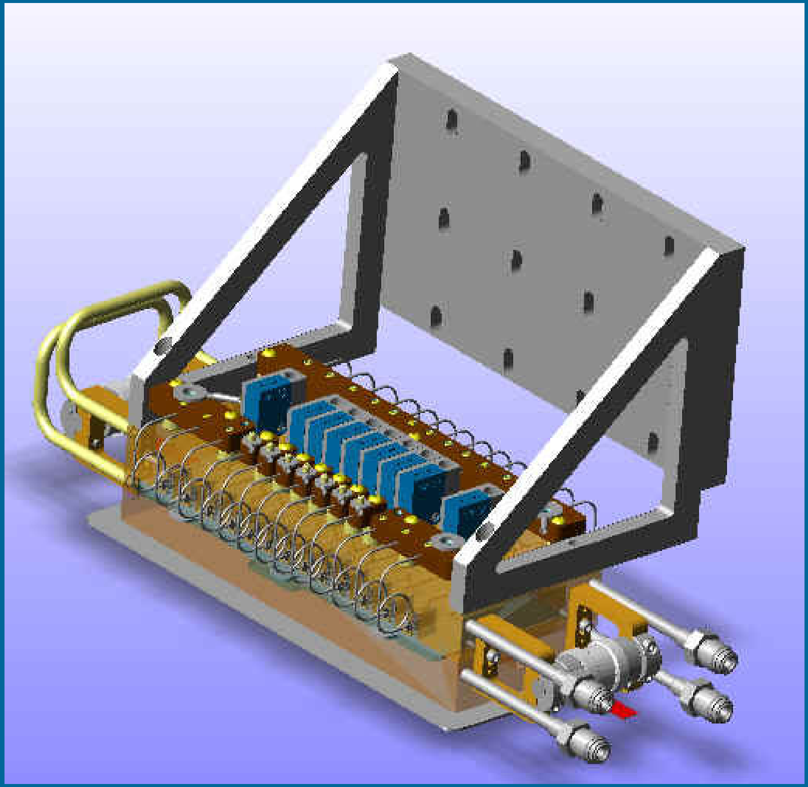
Key Issues for Higher Deposition Rates

- The envelope of processing conditions in PLD is strongly interrelated
- Significantly more stringent processing control is required for continuous deposition at higher rates



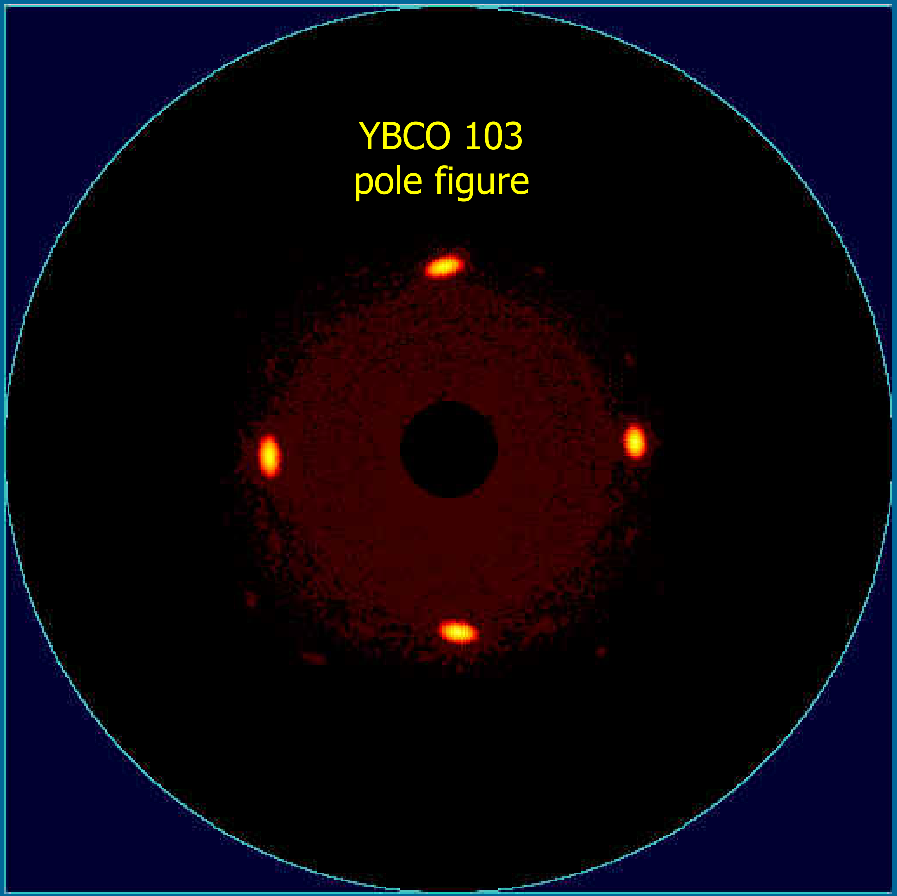
Heater Development

- To reach higher instantaneous rates temperature control is critical
- Implementation of a 9-zone quartz lamp-based heater
  - Temperature variation across the deposition zone
- We expect to achieve a more robust process for higher rates/longer lengths



Alternative Architecture

- Is it possible to deposit YBCO directly onto Epi-MgO/IBAD-MgO/Nickel alloy?
- We've seen good results on a number of different buffer layers
- If the IBAD/Epi layer is good (and thick) enough, do we need the buffer layer?



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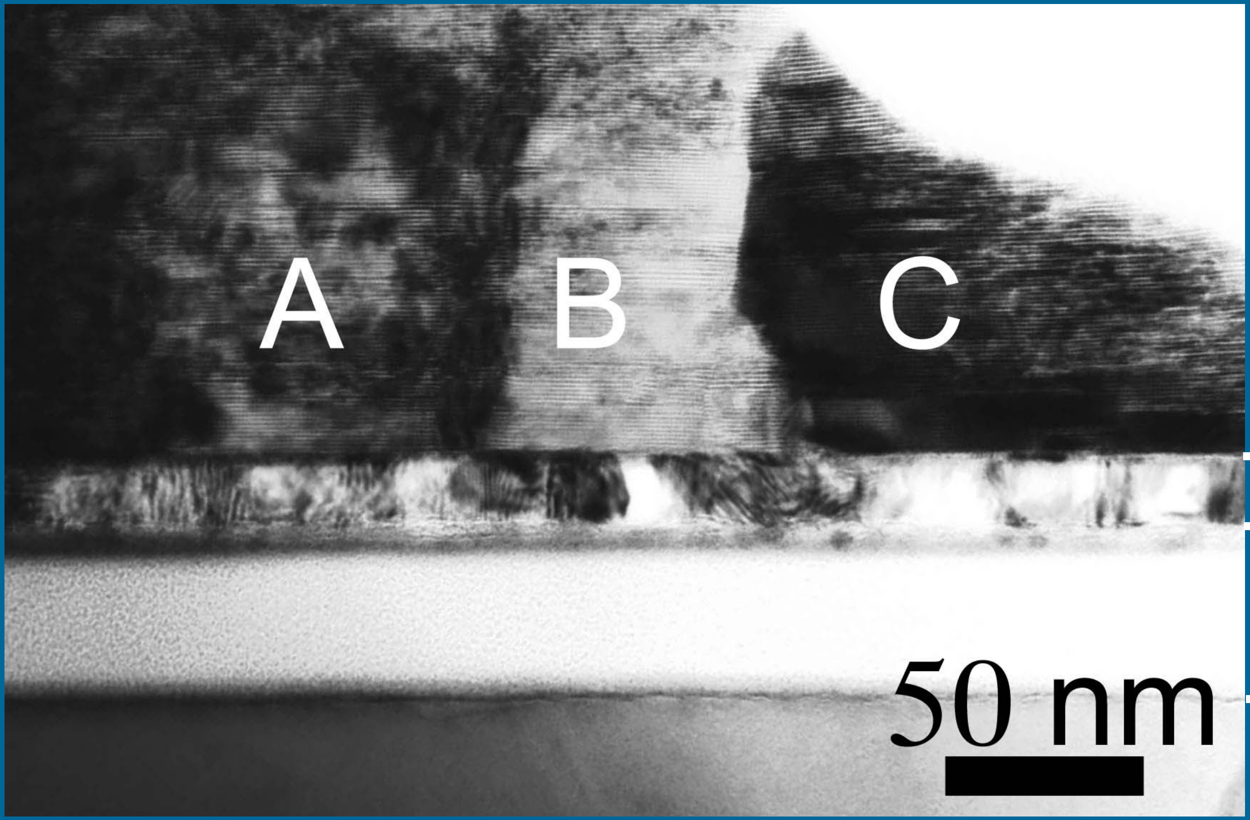
Microstructure



YBCO

IBAD MgO + Homo-epi MgO

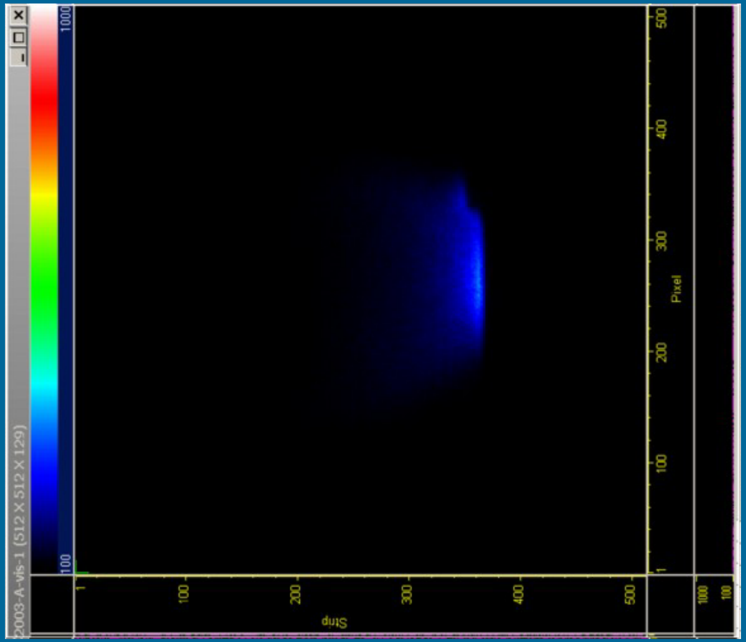
Seed Layer

Hastelloy C-276

- 0.5 μm thick YBCO deposited on homo-epi MgO/IBAD-MgO/Hastelloy C-276

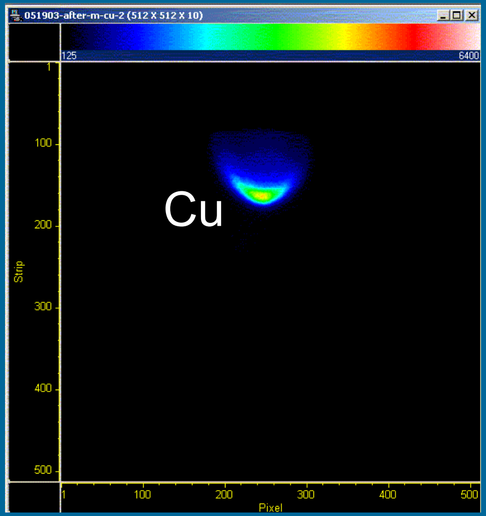
Collaborations

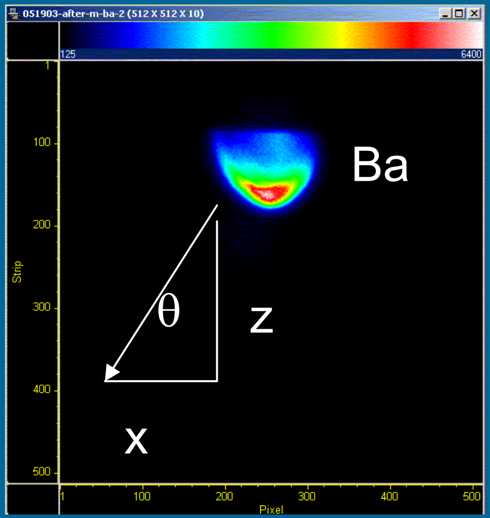
YBCO plume imagery



- With the Air Force Institute of Technology (Dr. Glen Perram and Carl Druffner)
  - Using a fast CCD camera and emission spectroscopy for plume analysis
  - Observe shield/plume interactions
  - Using filters, observe stoichiometry distributions within plume
    - Forward peaking of constituents

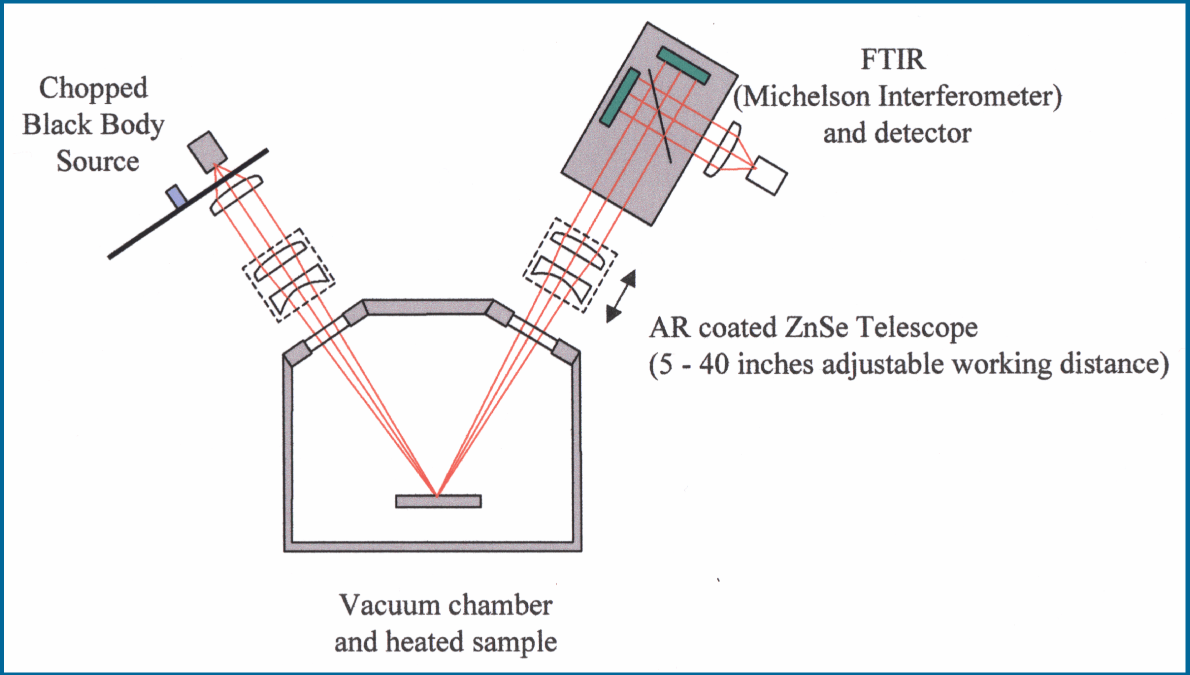
Collaborations





- With the Air Force Institute of Technology (Dr. Glen Perram and Carl Druffner)
  - Using a fast CCD camera and emission spectroscopy for plume analysis
  - Observe shield/plume interactions
  - Using filters, observe stoichiometry distributions within plume
    - Forward peaking of constituents

Collaborations



- With Stanford University (Bob Hammond and Gertjan Koster)
  - Using Fourier Transform Infrared Spectroscopy for substrate temperature measurement

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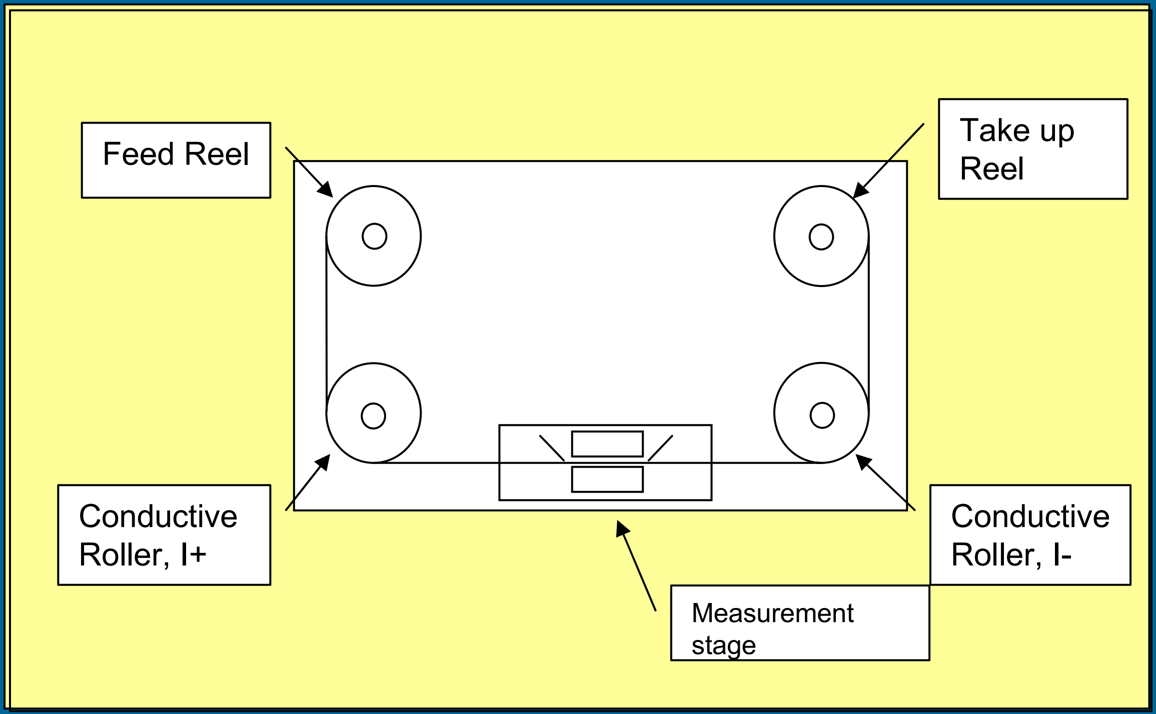
Continuous Critical Current Measurement System for Long Coated Conductors



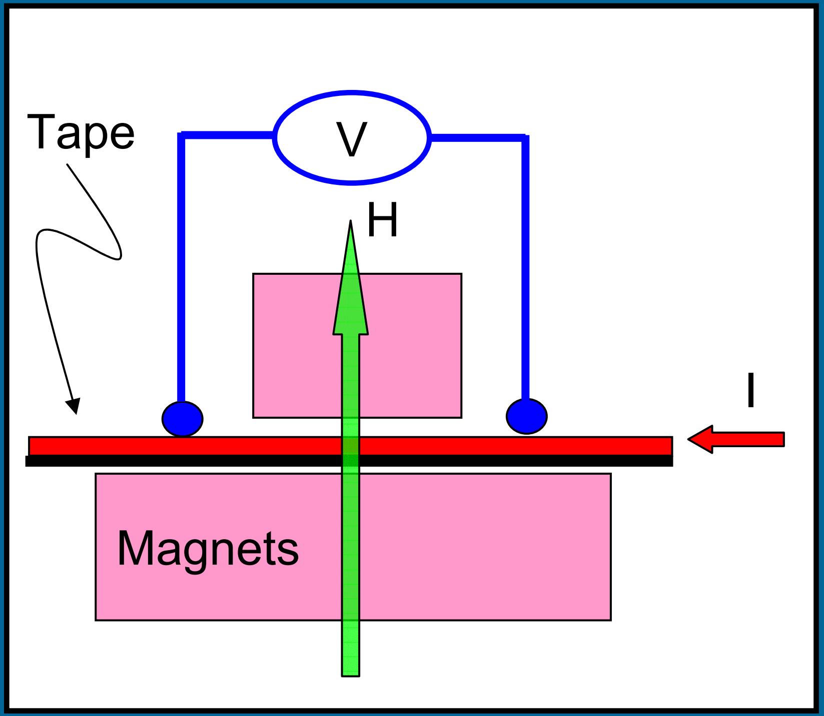
- Both for routine characterization and combinatorial research
- Tapes up to 10's of meters can be measured; operation and data acquisition fully automatic
- Tape transport mechanism
  - Resolution: 0.1 mm
  - Reproducibility < 0.25 mm for meter long tape

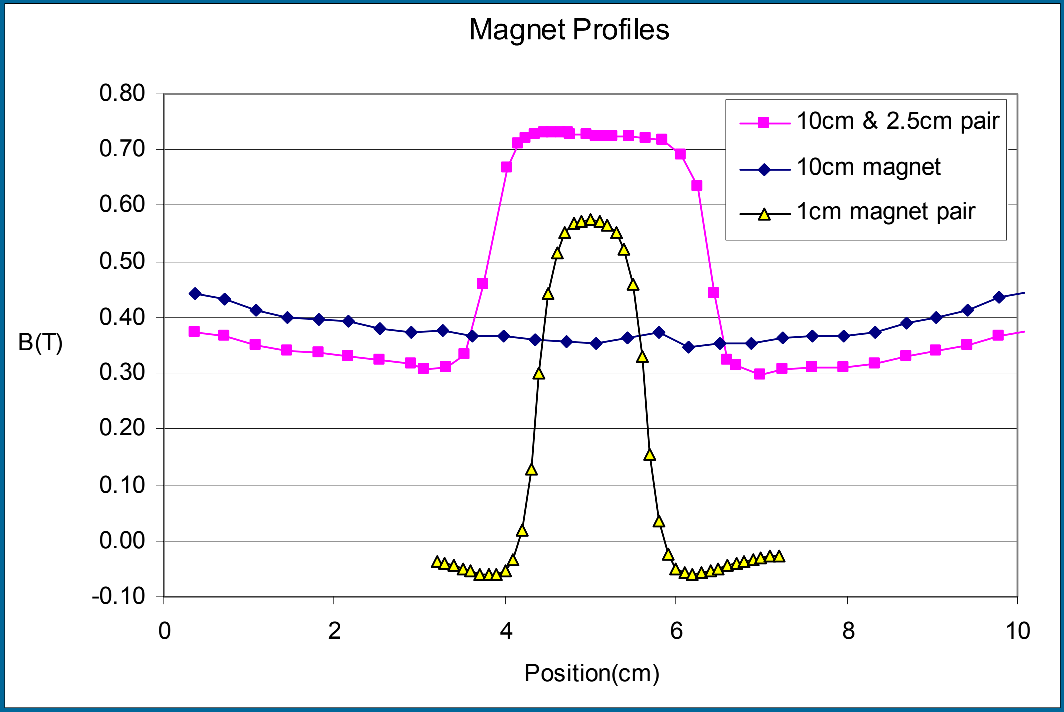
Transport measurements:

- Current contacts: copper wheels ~ 40 cm apart
- In-field measurements avoid sample damage
- Measurements made up to ~ 80 A
- Spring loaded voltage contacts, variable separation up to 30 cm
- Measurement rate: ~ 1 I-V curve/minute

Los Alamos

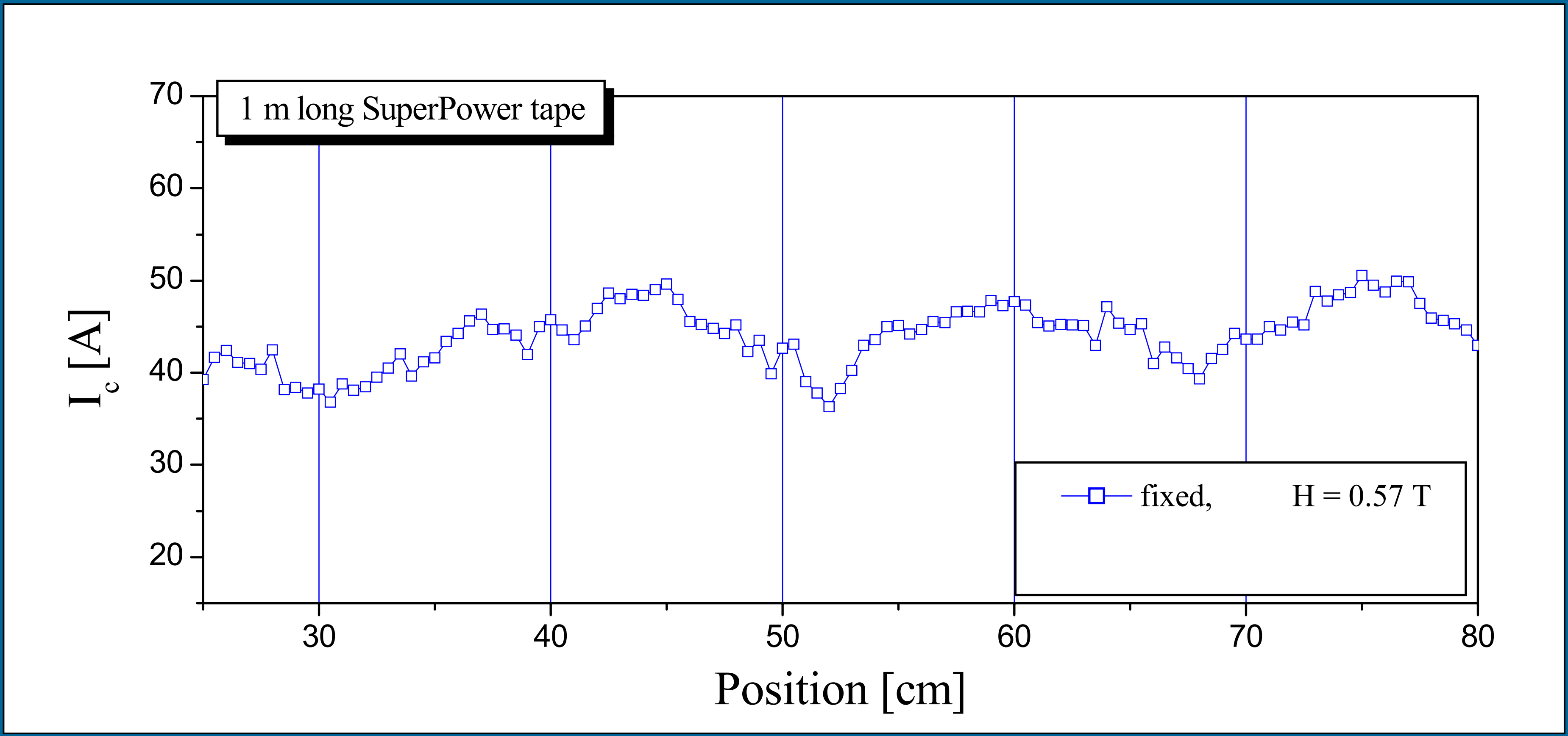
In-field transport measurements





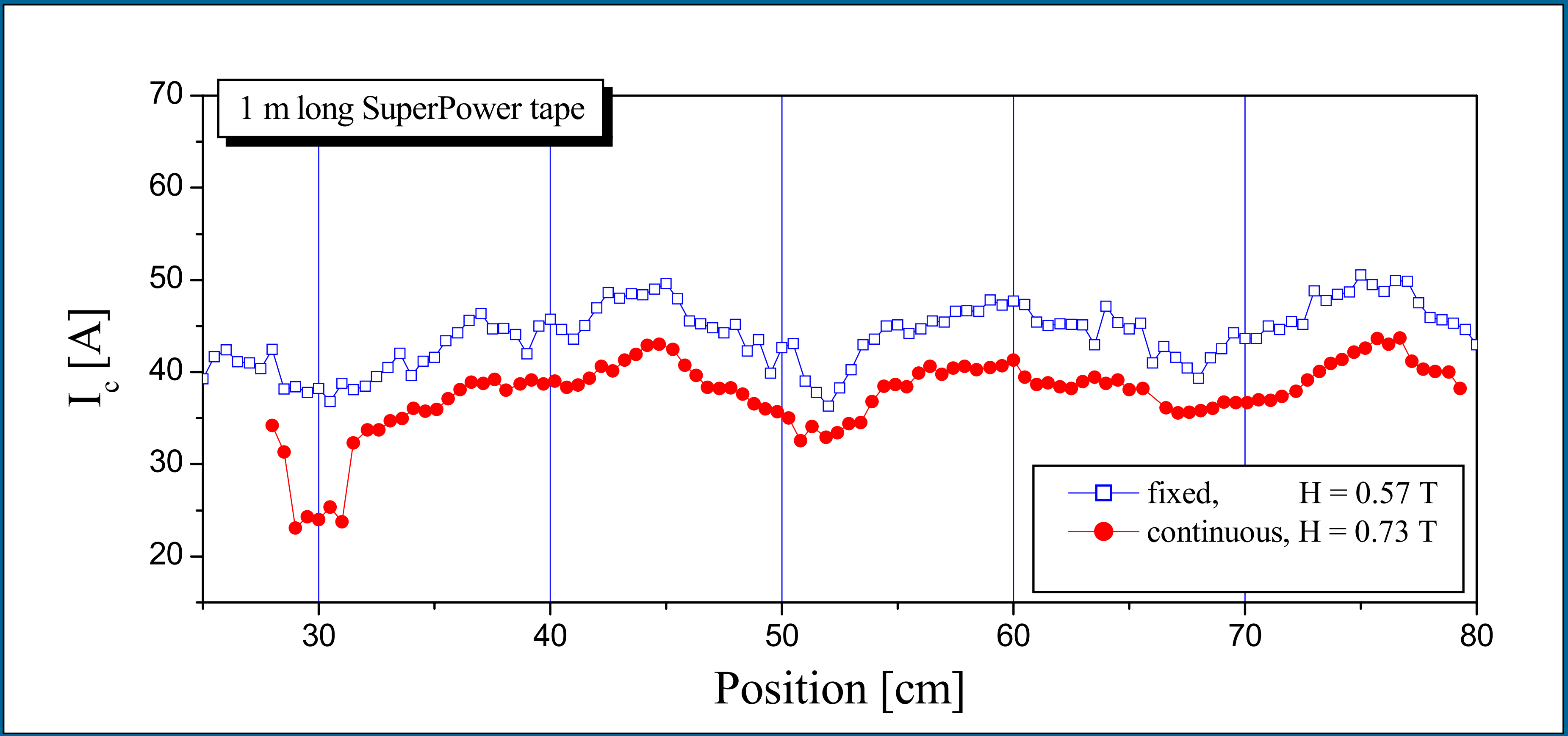
- Interchangeable permanent magnet setup allows flexibility in selection of field profile
- Fields up to 0.75 T available (reduction factor from self field ~ 6-7)

Comparison with measurements in a fixed-tape / sliding voltage contacts system



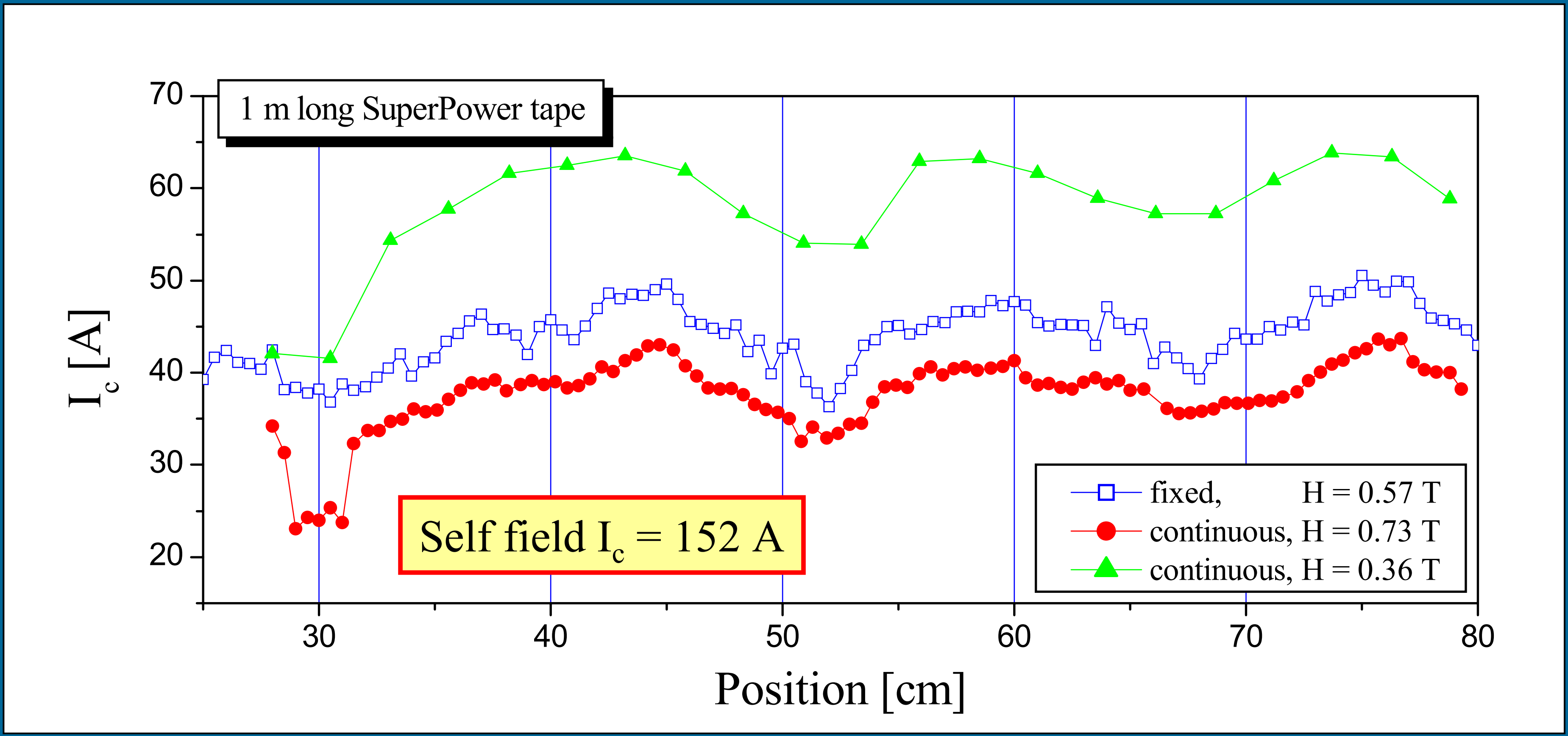
- Fixed-tape system designed for ~ 1 m long tapes, current flows end-to-end.

Comparison with measurements in a fixed-tape / sliding voltage contacts system



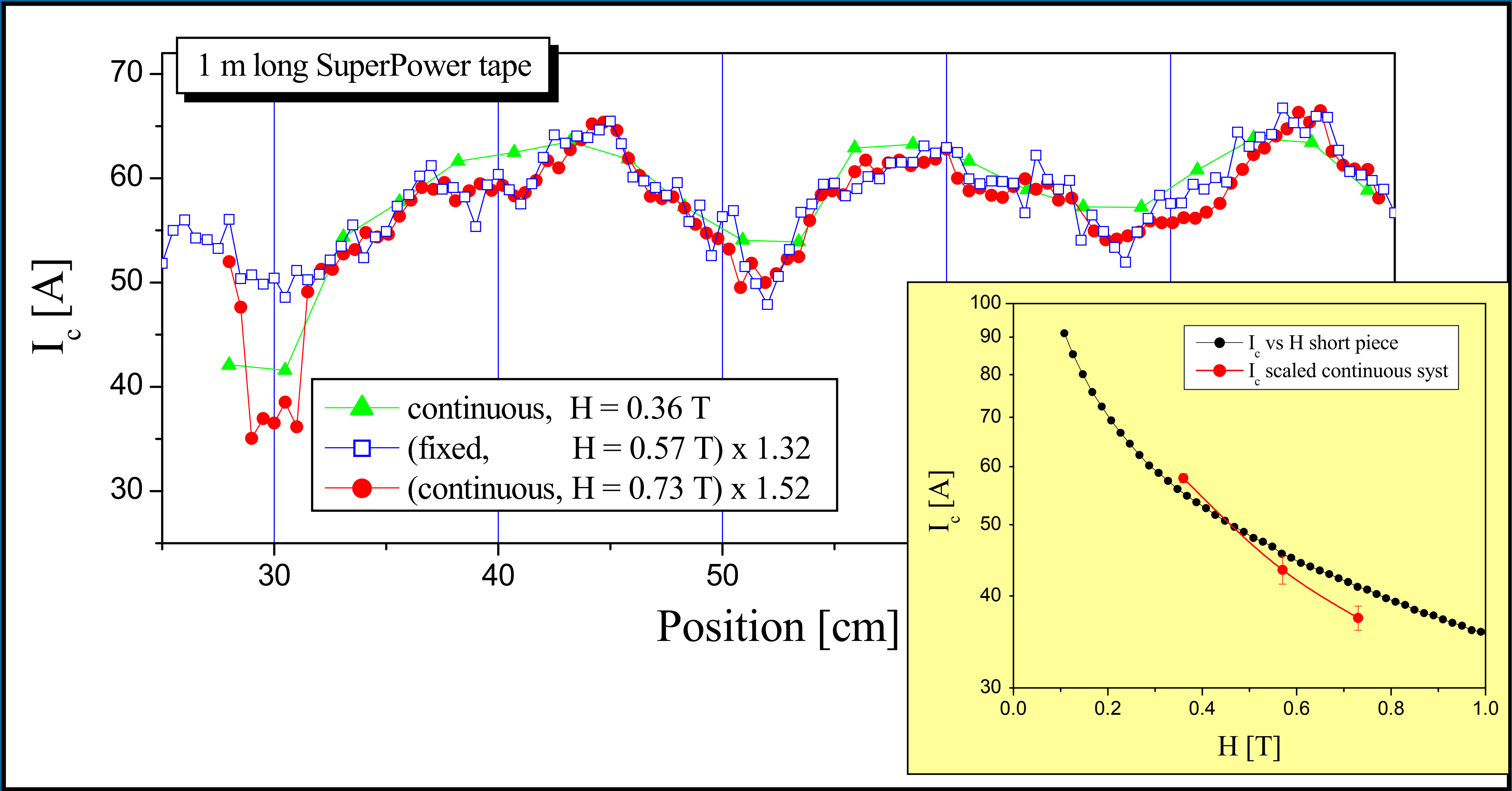
- Good agreement between both sets of data

Comparison with measurements in a fixed-tape / sliding voltage contacts system



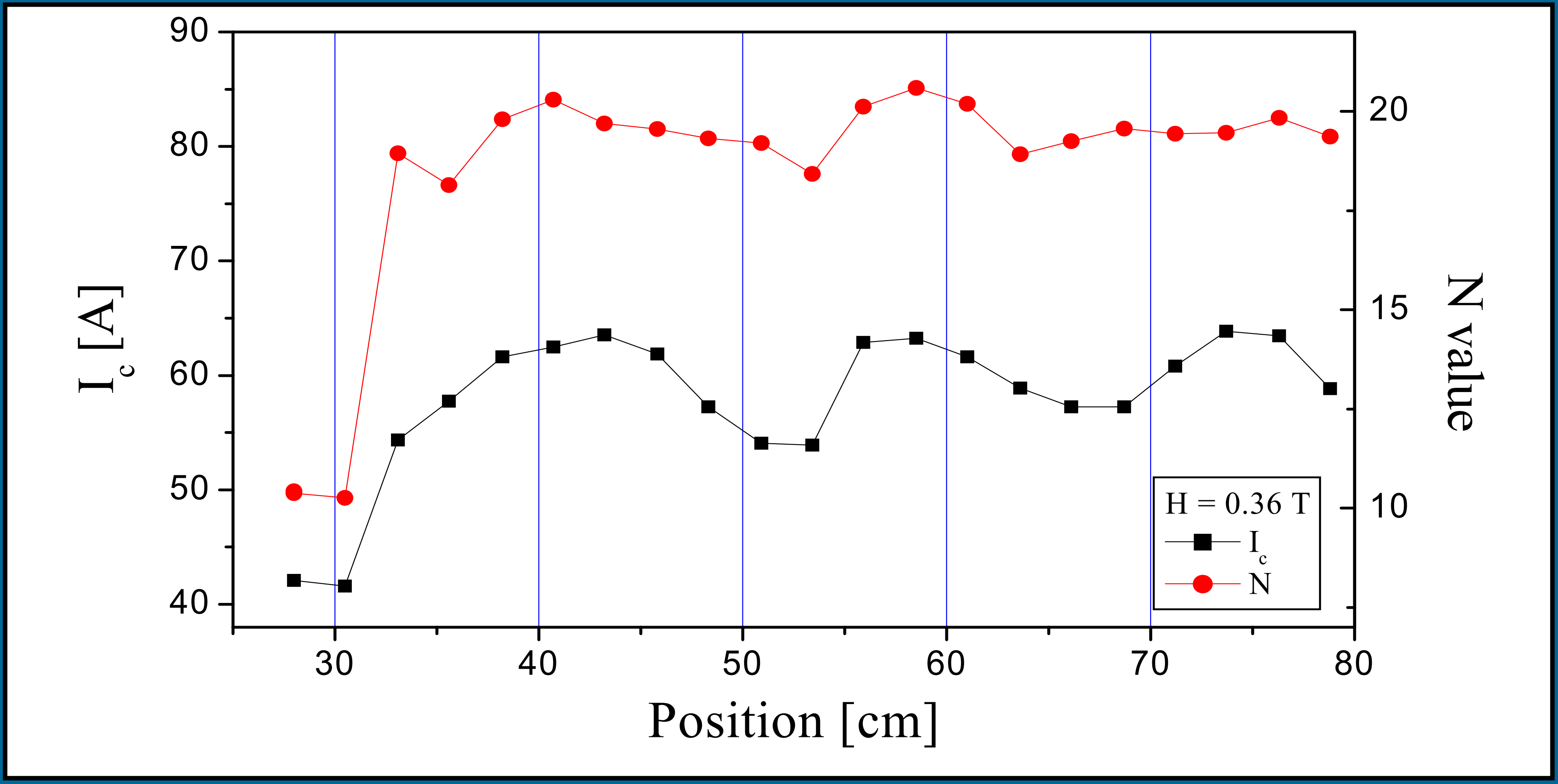
- Good agreement between all sets of data
- Speed (H=0.36T): 1.5 m/hr (2.5 cm per move)

Analysis of the field dependence



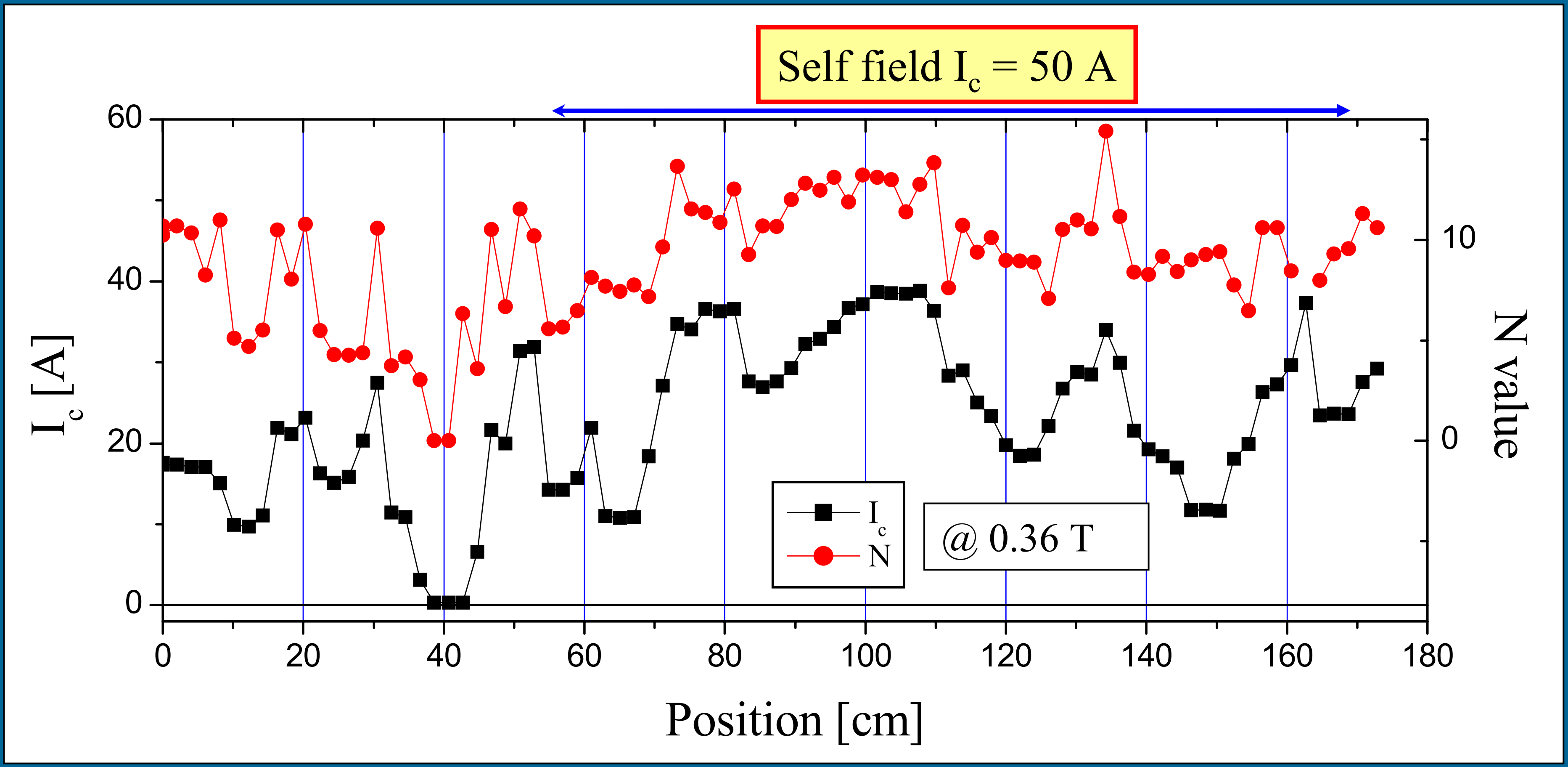
- Continuous in-field measurement provides required data without damage risk

The N values of the I-V curves correlate with I_c



- Inhomogeneities are at a microscopic scale

Results from first 2 m long tape produced at the LARP



- Nondestructive measurement possible even though one spot has I_c = 0
- Results used to select portion for self field end-to-end measurement

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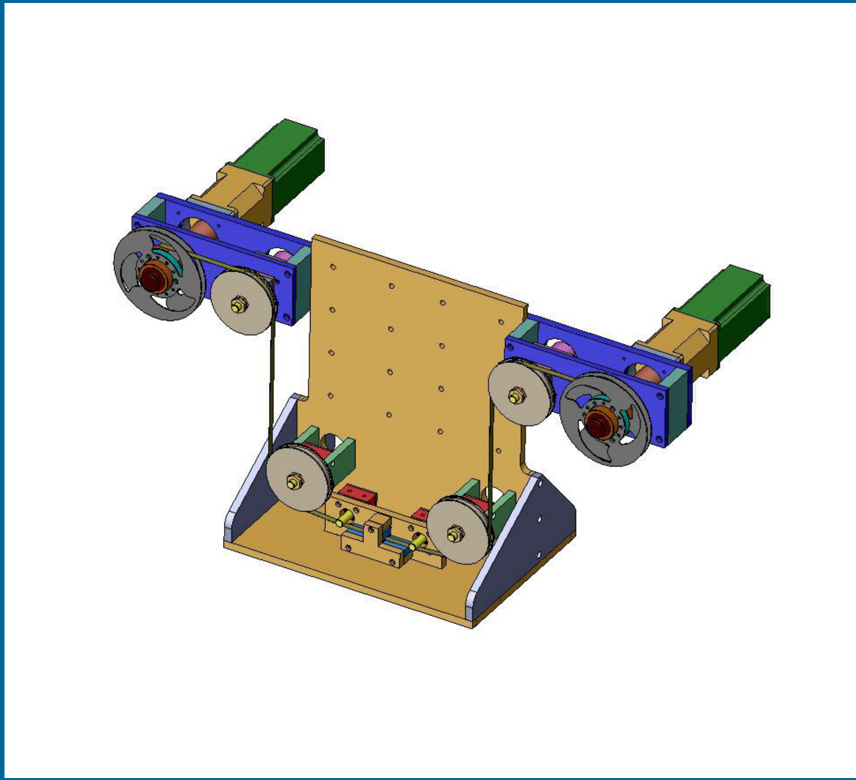
Development of a new system



Advantages:

- Tape load/unload at room temperature
- Can be inserted in fabrication line
- Adjustable separation between current contacts

Summary

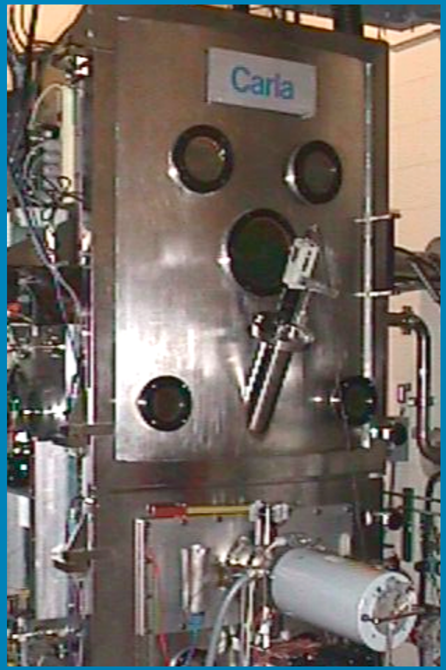
- Continuous critical current measurement system for long $YBa_2Cu_3O_7$-Based Coated Conductors has been built, tested and used for sample characterization
- In-field measurement provides required data without risk of sample damage
- Tapes up to 2 m long have been measured
- New, improved model under construction

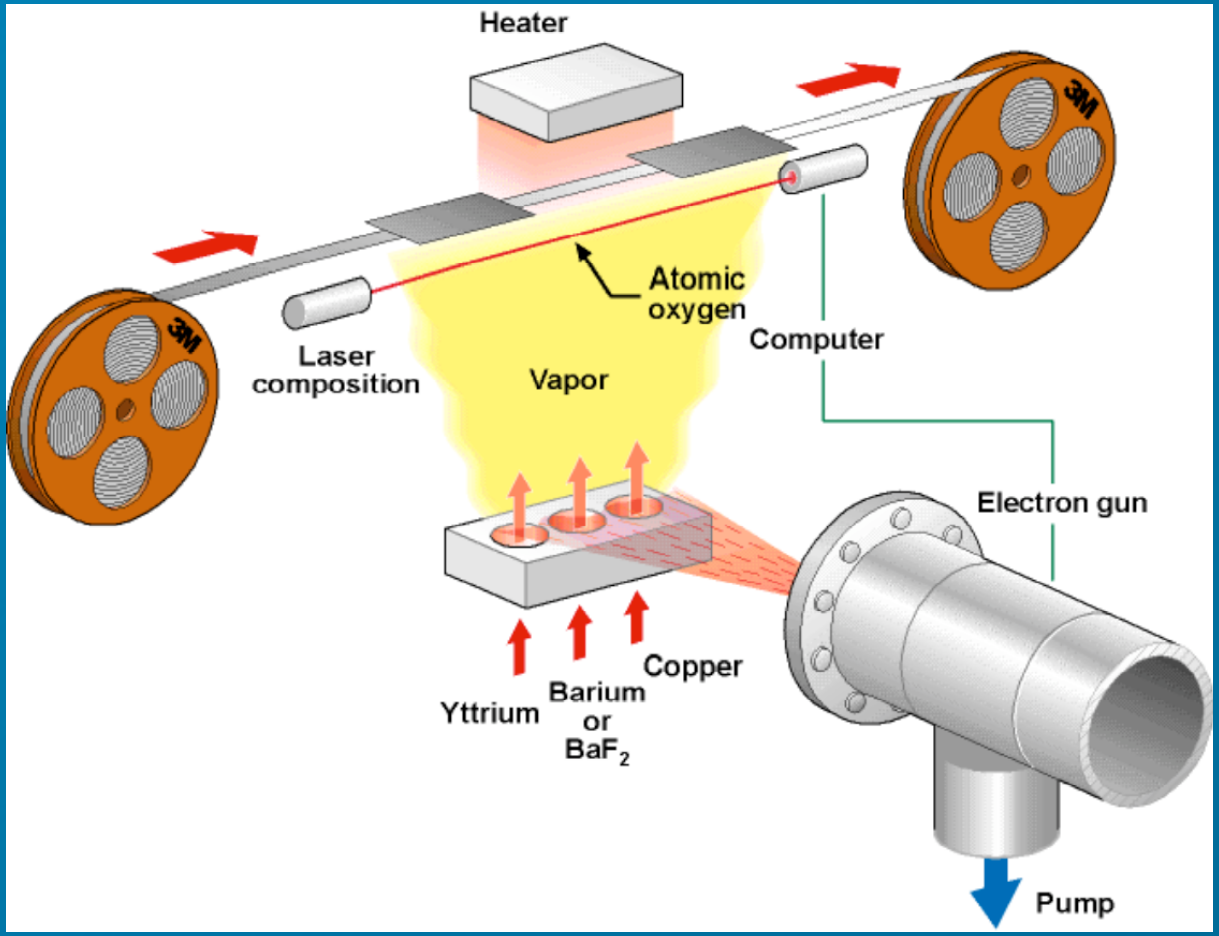
YBCO Deposition Processes

- PLD has demonstrated highest I_c's to date (Göttingen, Fujikura, LANL); best results are over 400 A/cm-width
- PLD is easiest to set up quickly and very versatile
- We want to explore other YBCO deposition processes, especially *in situ* deposition
- Reactive co-evaporation could be a good candidate for inexpensive CC production
- Reactive co-evaporation has been the workhorse for HTS electronics

Co-evaporation system to be installed at the Los Alamos Research Park

- e-beam evaporator with computer controlled scanning
- Laser-atomic absorption spectroscopy for accurate rate control
- Uses inexpensive source materials





Performance - 2003 Research Park Goals for CC Fabrication & Characterization

- **Goal: Complete the system integration and process control on fabrication systems - set up for ease-of-use and sample tracking**
  - new inspection station set up for long length tape examination
  - controls for automation being set up
  - tape handling systems set up and continually improved
  - goal partially complete

- **Goal: Achieve performance of IBAD-MgO coated conductors: MgO in-plane texture < 8° and I_C >50 A on 1-cm wide, long-length (> 5 m) tape**
  - IBAD MgO is now in-plane 6-7° routinely, with best cases 4-5°
  - long lengths >5 meters have been made with 6° [illegible]-FWHM; longest continuous IBAD was 12 meters
  - RP PLD has produced >170 A in short pieces and 50 A >1 meter

Performance - 2003 Research Park Goals for CC Fabrication & Characterization

- **Goal: Add ion scattering capability to *in situ* diagnostics**
  - system is ordered
  - working with the supplier for optimum design; performed initial measurements
  - scheduled for installation at LARP within 3 month
- **Goal: Establish a User Facility Program with a User Advisory Committee, incorporating at least 5 outside members**
  - Research Park is now established as a DOE User Facility
  - User Advisory Committee is established with the first chair Philip J. Pellegrino

Results - 2003 Research Park

- Bipolar electropolishing set up; producing < 1 nm RMS roughness over 100 meter lengths
- High rate IBAD-MgO at 36 m/hour; result showed 4.5° in-plane FWHM
- IBAD repair has been demonstrated (proof-of-principle)
- YBCO has in-plane texture of ≤ 3° FWHM routinely
- AMSC has made 98 A MOD-CC on a short piece of RP IBAD-MgO & Core Program (PLD) has made **250 A** over 20 cm on RP IBAD-MgO
- Continuous measurement of I_c positional dependence over 2 meter CC

Research Integration - 2003 Research Park

- Provided hundreds of meters of electropolished tape to industrial partners
- Provided meters of IBAD tape to partners
- Ongoing collaborations with AMSC and SuperPower, with a number of on-site visits
- Ongoing collaborations with AFIT, Stanford and national laboratories

Goals for FY 2004 - Research Park

- Reduce I_c variation to < 10% on a 2-cm measurement length scale over > 1 m
- Fabricate CC > 5 m with I_c > 200 A @ 75 K (J_c > 1 MA/cm^2)
- Provide IBAD-MgO to collaborators in lengths > 10 m with □□ < 8°; work with Core Program and industry to tailor CC architectures needed for the different YBCO processes
- Add ion scattering capability to IBAD processing system; utilize for diffusion barrier optimization
- Examine the IBAD repair in more detail and optimize HTS layers by maximizing I_c across the repaired regions (> 80% of average I_c)
- Implement YBCO reactive co-evaporation for CC; goal to make a superconducting 1 m length with 100 A @ 75 K

Summary

- Electropolishing producing nm-scale smooth tape over 100 m
- IBAD producing 10 m lengths of template tape (~ 6 - 8°)
- IBAD demonstrated at 36 meters/hour
- IBAD repair demonstrated with seamless transitions
- PLD producing CC meters
- TOF-ISARS surface analysis to be installed in the next months
- Co-evaporation of YBCO to be included in the next year